Vol. XIX May, 1942 No. 3

CEREAL CHEMISTRY

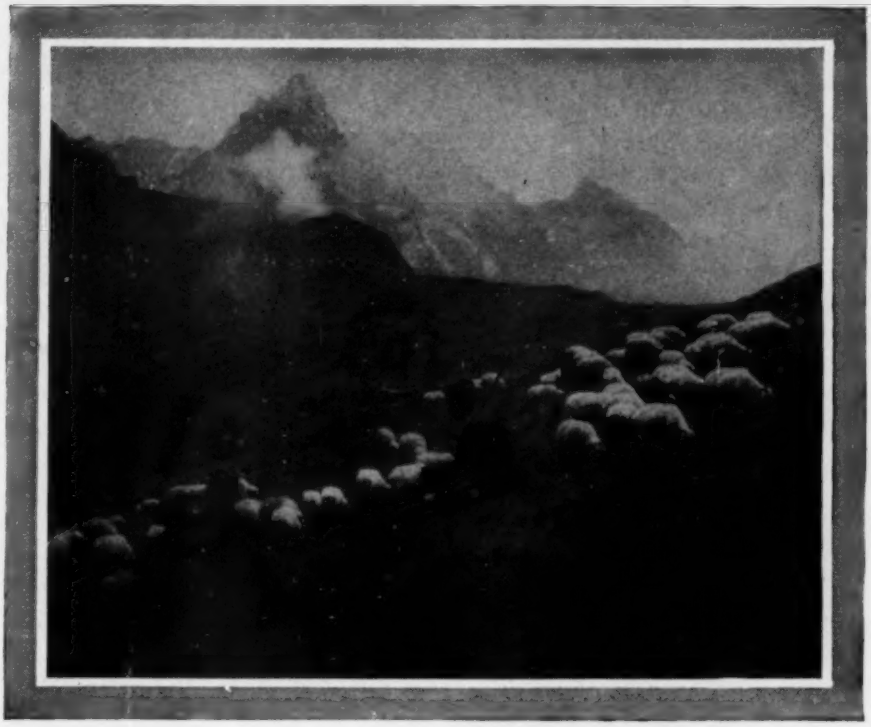
Published bi-monthly by the American Association of Cereal Chemists at Prince and Lemon Sts., Lancaster, Pa.

CONTENTS

Manuscripts for publication should be sent to the Editor-in-Chief. Advertising rates may be secured from, and subscriptions placed with the Managing Editor, Prince and Lemon Sts., Lancaster, Pa., or Agricultural Experiment Station, Lincoln, Nebraska. Subscription rates, $6 per year. Foreign postage, 50 cents extra. Single copies, $1.25; foreign, $1.35.

Entered as second-class matter at the post office at Lancaster, Pa., under the act of August 24, 1912.

Acceptance for mailing at special rate of postage provided for in Section 1103, Act of October 3, 1917, authorized February 16, 1924.



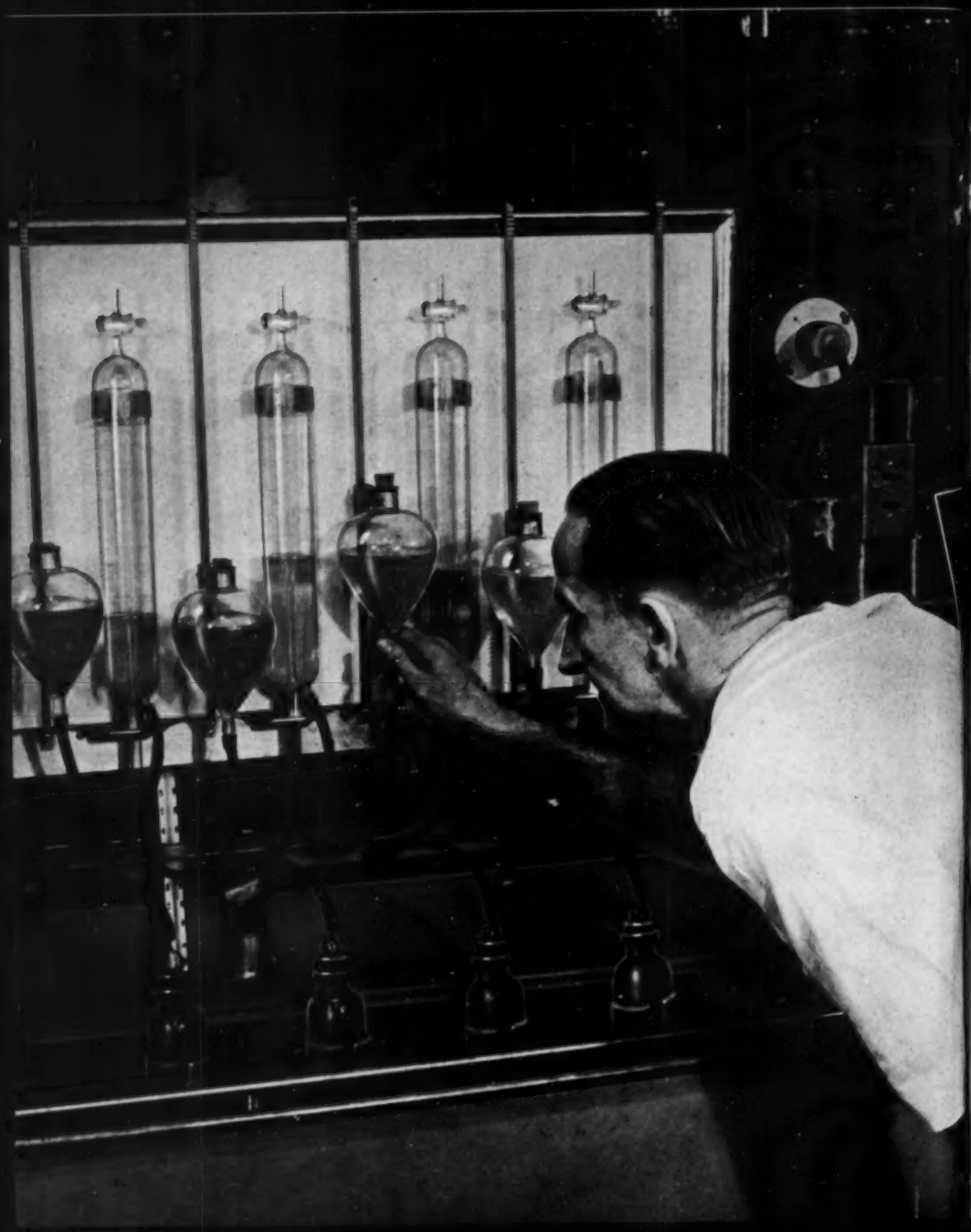
CONTRAST IN NUTRITION

Feeding 200,000,000 people engaged in total war presents a mighty problem. They cannot be herded on some choice mountain pasture and left to feed off nature's table. Food must be transported to them, at home and in distant lands, over facilities sorely taxed by rubber shortages, ship sinkings, and movements of military forces and equipment. To do this and at the same time safeguard against the vitamin deficiencies encountered even before the war, each cubic foot of space must carry new high vitamin values. Enriched flour will play a vitally important role in accomplishing this, especially since its thiamine and niacin levels hold up under widely varying climates. Moreover, the costs of enriching ingredients are down to new low levels. Time is precious. If your plans are not complete for "all out" enrichment of your white flour output get the advice of our technical staff without delay.

VITAMIN DIVISION

HOFFMANN-LA ROCHE, INC. · NUTLEY, N. J.

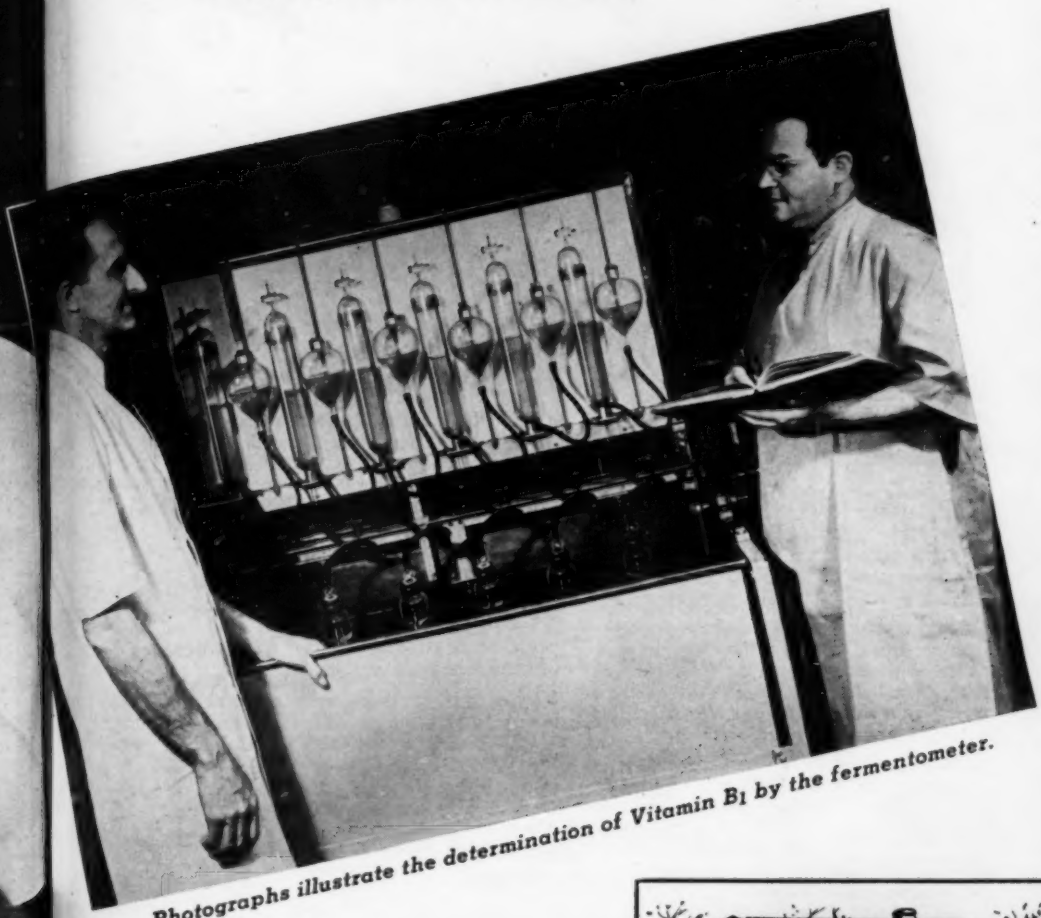
Helping the Baker help



FLEISCHMANN LABORATORIES

make America strong . . .

FOR MANY MONTHS one of the primary functions of the Fleischmann Laboratories has been research in connection with the development and improvement of Enriched White Bread . . . that product which represents the baker's tremendously important contribution to the success of the national program for better nutrition.



Photographs illustrate the determination of Vitamin B_1 by the fermentometer.

STANDARD BRANDS *Incorporated*

Makers and Distributors of Fleischmann's Yeast · 595 Madison Avenue, New York, N. Y.

Enrichment of cereal foods can be involved, tricky, difficult—or it can be simple as two-plus-two-equals-four. Why not keep it simple?

In "B–E–T–S"—our unique enrichment tablets for use in flour products—we have provided the easiest kind of enrichment method, one that is dependable and economical as well.

"B–E–T–S" contain vitamin B_1, niacin and iron in approved proportions—involve no change of formula or procedure—assure minimum "enrichment losses" in baking. "B–E–T–S" *retain potency well over a year when stored under normal conditions.*

When used for bread-enrichment, one "B–E–T–S" tablet added for every 100 lbs. of flour supplies, in each baked loaf, correct amounts of all three nutrients to meet minimum "enrichment" requirements. "B–E–T–S" are scored to break easily into quarter-sections for extra accuracy and economy when used in small doughs, parts of doughs, or with flour of high vitamin B_1 content.

Such *flexibility*, combined with other outstanding advantages, makes "B–E–T–S" your logical choice for easy, accurate, economical adjustment of nutrient-potency levels.

Write for further information and latest price-schedules. Stocks of "B–E–T–S" and "CRYSTALLINE B_1 WINTHROP"—crystalline thiamine hydrochloride, U.S.P.—are always ready at convenient points for quick delivery.

WINTHROP

—FOR CEREAL FOOD ENRICHMENT

ADDRESS INQUIRIES TO—

Special Markets Division

WINTHROP CHEMICAL COMPANY, INC.

170 Varick Street, New York, N. Y.

NEW BECKMAN

PORTABLE pH METER

Quinhydrone Electrode

21040

The instrument is mounted within a carrying case for easy portability and may be placed in operation by merely opening the case, inserting the electrodes and immersing them in the test liquid contained in a beaker. The instrument is mounted to slide up or down on a rod attached to the inside of the carrying case, so no other support stand is required. If desired, the instrument may be removed from the case for direct immersion of the electrodes into vats, etc. The indicating meter, located on the top of the instrument, has a range from 0 to 9 pH with 0.1 pH graduations. A multiple scale is provided in 5° C steps for the temperature range from 0 to 50° C, so that direct temperature-corrected pH readings may be made without resort to correction tables or calculations. A magnifying lens facilitates reading the pH scale.

Dimensions of case: Height, 11½ inches; width, 7 inches; depth, 4½ inches. Complete with carrying case fitted with lock and carrying handle, gold and calomel electrodes, 30 ml beaker, metal-case thermometer of 0° to 50° C range, magnifying lens, 3 grams guinhydrone crystals, 20 grams KCl crystals, and dropper bottle of KCl solution.

21040 HYDROGEN ION METER, Beckman Quinhydrone.....Each $75.00

CENTRAL SCIENTIFIC COMPANY

SCIENTIFIC INSTRUMENTS — TRADE MARK CENCO REG. U.S. PAT. OFF. — LABORATORY APPARATUS

New York • Boston • CHICAGO • Toronto • San Francisco

A SYMBOL OF LEADERSHIP

in the Pure Vitamin Field

Ever since the first of the pure vitamins (ascorbic acid) was synthesized in 1934, the name Merck has been identified with leadership in the synthesis, development, and production of these vitally important substances.

The growing list of Merck contributions in this field emphasizes the outstanding rôle being played by Merck chemists and their collaborators in making available pure vitamins of known and uniform potency.

As the foremost manufacturer of pure vitamins, Merck & Co. Inc. offers the food processor an established and dependable source of these essential nutrients.

Backed by thorough experience, extensive resources, modern and rapidly-expanding production facilities, Merck is well qualified to serve food processors who are preparing to improve their products through the addition of pure vitamins.

Our scientific staff and laboratories are prepared to serve you.

MERCK PURE VITAMINS

VITAMIN B_1
(Thiamine Hydrochloride)

RIBOFLAVIN
(Vitamin B_2)

NIACIN (Nicotinic Acid)

NIACIN AMIDE
(Nicotinic Acid Amide)

VITAMIN B_6 HYDROCHLORIDE
(Pyridoxine Hydrochloride)

CALCIUM PANTOTHENATE
DEXTROROTATORY

ASCORBIC ACID
(Vitamin C)

VITAMIN K_1
(2-Methyl-3-Phytyl-1, 4-Naphthoquinone)

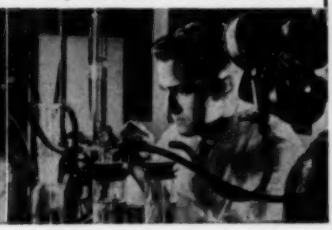
2-METHYL-NAPHTHOQUINONE
(Menadione)
(Vitamin K Active)

ALPHA-TOCOPHEROL
(Vitamin E)

Isolation of a Vitamin

Chemical Assay of Vitamin B_1

Analytical Test in Vitamin Procedure



MERCK & CO. Inc. *Manufacturing Chemists* **RAHWAY, N. J.**

New York • Philadelphia • St. Louis

In Canada: MERCK & Co. Ltd., Montreal and Toronto

TWENTY-EIGHTH ANNUAL CONVENTION AMERICAN ASSOCIATION OF CEREAL CHEMISTS

Edgewater Beach Hotel
Chicago, Illinois
May 18 to 22, 1942

Alertness to the problems confronting chemists in the food industries is your responsibility during the emergency. It is your duty to attend your convention for first-hand information.

PROGRAM FEATURES

Osborne Medal Award Address
Statistics in Cereal Chemistry
An Address on "Starch"
Protein Symposium
An Address on "Enzyme-Vitamin Relationships"
An Address on "Water Soluble Vitamins"
Bread and Flour Nutritional Supplements

ADDITIONALS

Industrial Trips Hobby Show Equipment Exhibits
Annual Banquet Collectors Exhibit

President
C. N. FREY
The Fleischmann Laboratories
810 Grand Concourse, New York City, N. Y.

Program Chairman:
H. R. KRAYBILL
American Meat Institute
59 East Van Buren
Chicago, Illinois

Local Arrangements Chairman:
R. A. BARACKMAN
Victor Chemical Works
Chicago Heights, Illinois

CEREAL CHEMISTRY

VOL. XIX MAY, 1942 NO. 3

A VISCOMETRIC DETERMINATION OF THE OPTIMUM pH FOR THE PROTEOLYTIC ACTIVITY OF MALT WITH GELATIN AS A SUBSTRATE

JOHN R. KOCH and SISTER MARY LAURETTA, S.S.N.D.

Department of Chemistry, Marquette University, Milwaukee, Wisconsin

(Read at the Annual Meeting, May 1941)

Various methods are now available for the quantitative determination of the action of proteolytic enzymes. This is followed either by observing the amount of substrate digested at the end of some given time or by determining, at intervals, the changes in the viscosity, conductivity, or turbidity of the substrate occurring during the course of the digestion effected by the enzyme (Waksman, 1926). The hydrolytic breakdown of gelatin, for example, caused by the action of the proteolytic enzymes of malt shows itself through a noticeable decrease in the viscosity of the gelatin-enzyme mixture (Ehrnst, 1938; Koch, Nelson, and Ehrnst, 1939; Northrop, 1922). The report here presented is based upon a study carried out by following such a decrease in viscosity when the pH value of the substrate was made the variable factor.

The proteolytic enzymes in malt function differently under different pH conditions (Britton, 1929; Hagues, 1924; Hopkins and Kelly, 1931). Viscosity tests show that proteolysis can be either hastened or retarded as the pH is varied (Hind, 1938; Northrop, 1923), and the pH may be varied by changing the acidity or alkalinity of the liquid in which the enzyme is present or by changing that of the solution of the product upon which the enzyme is to act (Tauber, 1937; Van Laer, 1923). Waksman (1926) and Northrop (1922) state that most enzymes are greatly influenced by the reaction of the medium in which they act, and that there is an optimal H-ion concentration for the activity of each enzyme, which accounts for upper and lower limits of reaction, above or below which the enzyme is inactive or may be rapidly destroyed.

It is well known that pepsin becomes inactive in alkaline solutions because its activity is restricted to an acid medium only. Trypsin digests proteins in either neutral or alkaline solutions, but not in an acidified medium (Northrop, 1922). Malt contains several enzymes,

and consequently its activity is spread over a wide pH range. However, each enzyme of malt has its own zone, below or beyond which it is inactivated or completely destroyed (Hagues, 1924; Mill and Linderstrøm-Lang, 1927).

Attempts to determine these zones of maximum activity have been made and a number of methods have been applied (Britton, 1929; Hopkins and Kelly, 1931; Hopkins and Krause, 1937; Lüers and Malsch, 1929; Lundin, 1923; Mill and Linderstrøm-Lang, 1927). However, none of the literature studied by the authors offers any viscometric approach to finding the optimum pH for the proteolytic enzymes of malt. Yet there is substantial evidence that more attention should be given in that direction (Ehrnst, 1938; Kolbach and Simon, 1936).

In the brewing industry the clarification and maturing of malt beverages and the correcting of protein haze is almost wholly dependent on proteolytic enzymes. Moreover, the amylase activity, so vital in malting and brewing, also depends on the preceding proteolysis (Wallerstein, 1939).

The purpose of this research was to study the viscometric determination of the optimum pH for the proteolytic activity of malt with gelatin used as a substrate. This peak activity was obtained by permitting the digestion of the substrate to occur when the pH condition of the gelatin was altered, (*a*) by using HCl and NaOH as modifying agents (Figs. 3, 4, and 5), and (*b*) by adding buffers as stabilizing factors (Fig. 6). The malts analyzed were of the commercial variety supplied to breweries and the results presented are from a study of four malts.

The viscosity measurements followed the method developed by Koch, Nelson, and Ehrnst (1939). The instrument used was a new-type, all-glass digestion-flask viscometer.

Figure 1 shows the viscometer in detail. Not only does it meet requirements for low cost, simplicity of design and operation, and adaptability to routine work, but it is also reliably accurate, and conveniently permits the measurement of viscosity at the end of any desired period in order to observe the progress of enzymatic action (Koch, Orthmann, and Degenfelder, 1939; Koch, Nelson, and Ehrnst, 1939). This viscometer as originally designed was constructed in two forms. The first had its upper and lower reservoirs held together by means of two rubber stoppers; the second was an all-glass type, which later was replaced by a more sturdy model.

In detail, the present model is assembled with glass seals throughout and serves both as a viscometer and as an almost hermetically sealed digestion flask. Its capillary, about 35 mm long, is permanently sealed

in at the junction of two 200-ml Erlenmeyers. Because the diameter of the capillary is an important factor, the bore of the one selected was such that a working volume of 50 ml of distilled water had an efflux time of approximately 30 seconds. Longer efflux time was considered unsatisfactory because the drainage time between 15-minute runs was thus somewhat shortened, and a viscous substance such as gelatin re-

Fig. 1. Viscometer.

quires more time than less viscous liquids to drain completely from the walls of a glass container. Furthermore, a fixed funnel-shaped entrance to the capillary eliminated corrections for the retention of any liquid after a run was made, in contrast to the earlier viscometer used by Nelson. The externally sealed-in side arm of the viscometer not only allowed the confined liquid to flow from one reservoir into the other at the completion of a run, but it also made the handling and inverting of the viscometer an easy routine procedure.

The viscometers were calibrated by means of a 40% sucrose and a 25% glycerol solution. By using these solutions at 25°, 30°, and 40°C the calibration was made over a wide range and carefully checked. For the details of calibration either of two previous papers may be consulted (Koch, Orthmann, and Degenfelder, 1939; Koch, Nelson, and Ehrnst, 1939). For general calibration procedure the works of Sheely (1923, 1932), Bingham and Jackson (1916), Barr (1931), and Hershel (1917) should be consulted.

Preparation of Solutions

The method of preparing a 10% stock solution of gelatin was essentially the same as that described by Koch, Nelson, and Ehrnst (1939). From this supply the 6⅔% concentration was obtained by dilution.

The malt infusion was prepared at 40°C by adding 20 g of malt (finely ground in a Miag-Seck mill) to 100 ml of distilled water. This was kept in the water bath (at 40°C) for 30 minutes (being stirred frequently during the time), and then filtered. One 35-ml sample of the filtrate (attemperated in the water bath for about 8 minutes) was reserved for the digestion test.

The pH of the substrate was changed by selective additions of one of three agents: a buffer, HCl, or NaOH, added to the gelatin (10%) before it was made up to mark. Clark and Lubs' set of buffers was employed. These mixtures were made according to the procedure indicated by Clark (1923) and the acid and alkali were prepared in the usual laboratory way. Preliminary trials to determine the amount of buffer required to bring any particular digestion mixture to a desired pH were made prior to making any test runs. The pH of the substrate for malts 1, 2, and 3 was adjusted by adding 0.2*M* HCl solution or 0.2*M* NaOH. The buffer mixtures were added to the gelatin prepared for malt 4.

For each change of pH, a predetermined amount of buffer or acid or alkali was added to the gelatin that served as the 10% stock solution for the test under observation. The pH value recognized as the working pH of a run was the one obtained within the first five minutes after the malt extract was added to the gelatin. The pH, covering a series of tests for the malts selected, ranged from approximately 2.5 to 8.0.

Determinations and Results

Malt blank: About 35 ml of the extracted malt was placed into boiling water for 4 minutes, and then cooled rapidly. The precipitated albuminoids were filtered out and 25 ml of the filtrate was attemperated to 40°. Fifty ml of gelatin (previously prepared and attemperated) was now added to 25 ml of the boiled extract. Of this 6⅔%

mixture, 50 ml was transferred to a viscometer and run as a blank. Because the enzymes were inactivated by boiling (Koch, Nelson, and Ehrnst, 1939), the outflow time of the blank showed no appreciable variation (table showing blank for Fig. 2).

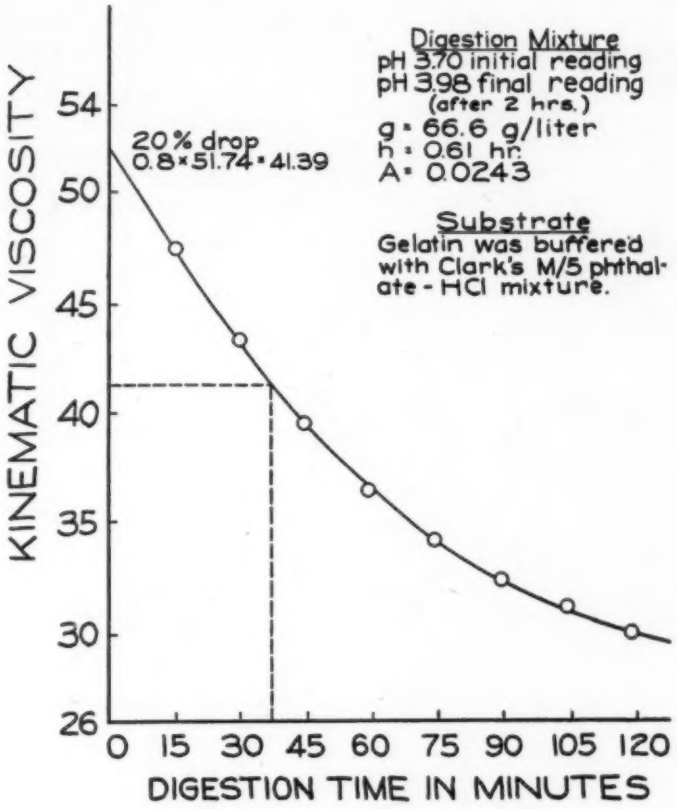


Fig. 2. Malt 4, pH 3.70.

Viscometer A—Blank

Time intervals (*min*)	Viscosity reading (*sec*)	K (*millipoise*)
15	61.4	54.03
30	59.7	51.63
45	59.2	50.92
60	59.4	51.20
75	59.2	50.92
Average	59.78	51.74

Viscometer B

Time intervals (*min*)	Viscosity reading (*sec*)	K (*millipoise*)
15	49.7	47.67
30	47.5	43.13
45	45.8	39.53
60	44.4	36.51
75	43.4	34.32
90	42.6	32.54
105	42.0	31.20
120	41.9	30.07

The purpose of running the blank was to establish an initial or zero point. Attempts at finding the value by extrapolating the curves resulting from the two-hour test runs proved satisfactory enough to be accepted. In fact, later in a personal conference with Mr. Lawrence Ehrnst, chief chemist of the Froedtert Grain and Malting Company, Milwaukee, Wisconsin, it was learned that he found extrapolation reliable enough for routine use in viscosity tests conducted in his

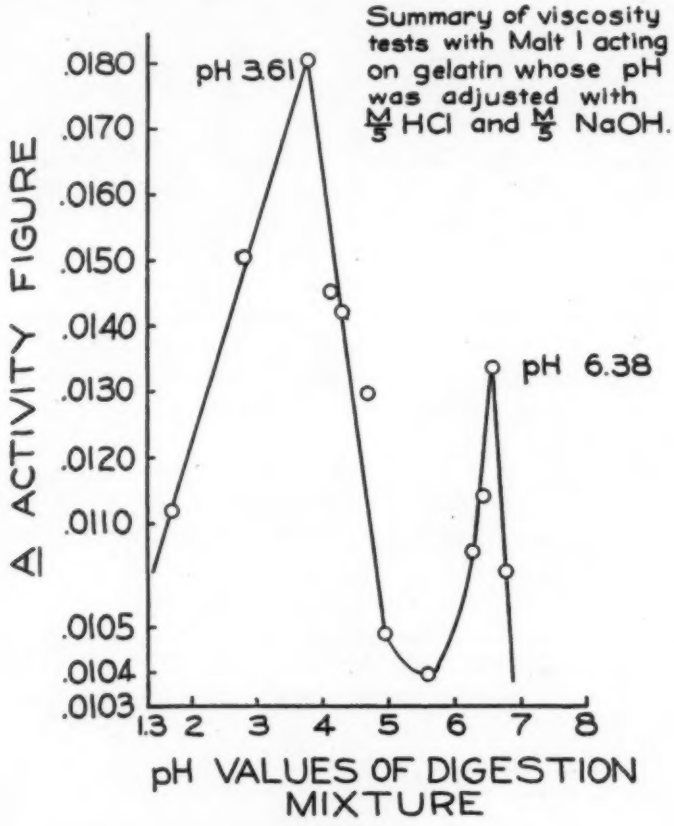


Fig. 3. Malt 1.

laboratories. A survey of viscosity work done by Northrop (1922, 1923) and more recently by Laufer (1937, 1938), both with gelatin as a substrate, confirms the extrapolation method for establishing a zero point. However, for the sake of strictly reproducible data as well as for the maintenance of uniformity, a blank was run in this study for every change of pH.

Viscosity determination: It was found by many comparative trials that the most efficient method of making viscosity determinations in

these experiments was to run the blank in one viscometer and the digestion mixture in another, so that both would be subjected to identical conditions of temperature and that both would have the substrate drawn from the same stock solution (freshly prepared for each change of pH), which in turn had had the same facilities for attemperation. Values were easily duplicated as a result.

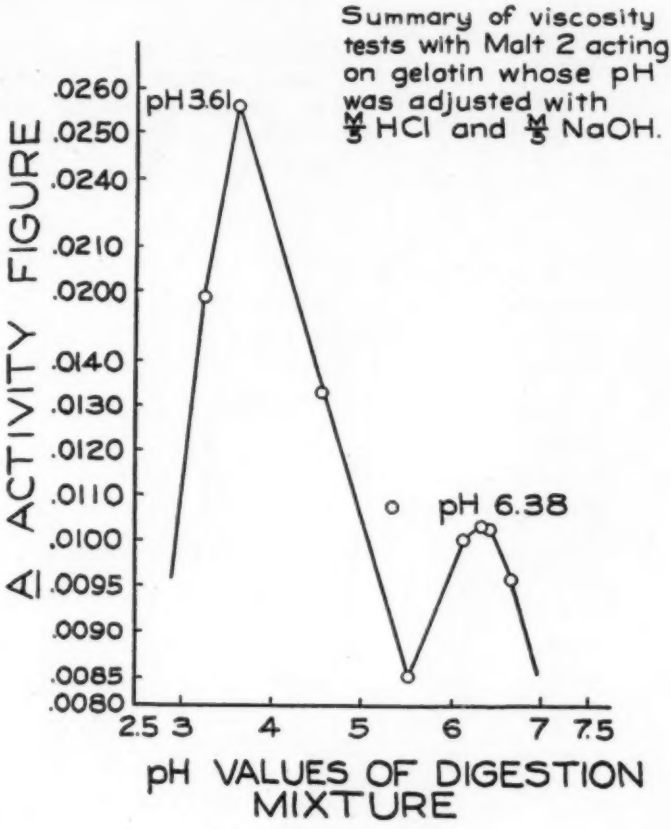


Fig. 4. Malt 2.

The time of adding 50 ml of the attemperated 10% gelatin-buffer mixture or gelatin-acid mixture to 25 ml of the unboiled extract (also at 40°) was considered the initial or zero time of the run, since digestion was begun from the moment the solutions came in contact with each other. Fifty ml of this digestion mixture was transferred to the one viscometer and 50 ml of the blank mixture was conveyed to the other. The viscometers were immersed in the water bath as soon as they were filled. At the end of 15 minutes, each viscometer was inverted in turn

and the time of flow for each was determined by timing the transit of the digestion mixture and the blank from the upper to the lower reservoir of the respective viscometer. At the completion of a run the viscometers were again put into their position of rest. Viscosity determinations were made every 15 minutes for two hours in nearly all cases for the viscometer containing the digestion mixture and for a period

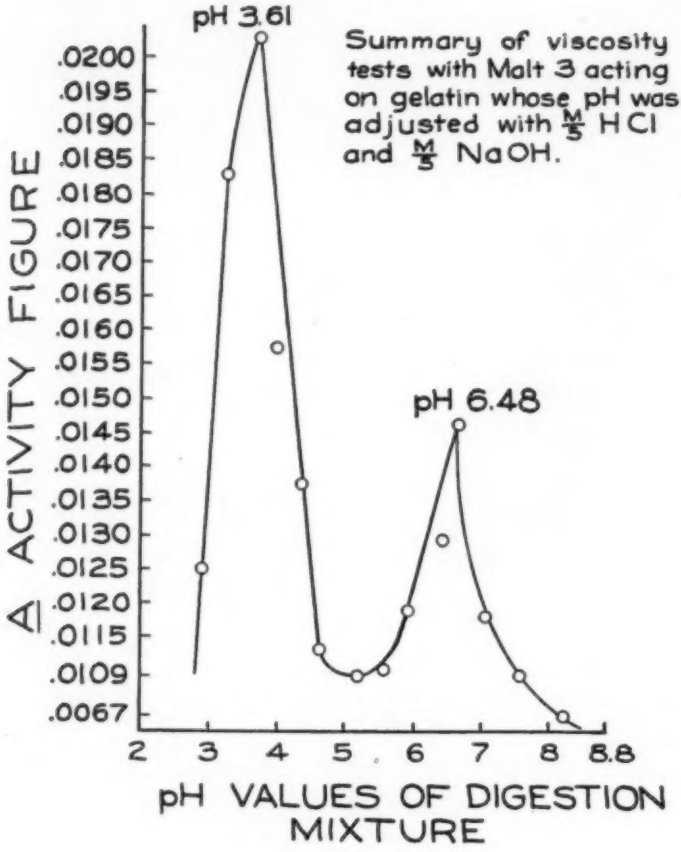


Fig. 5. Malt 3.

long enough to make five trials for the blank. An average of these five trials established the initial point when the viscosity values were graphed.

Figure 2 and the corresponding numerical representation indicate a series of runs obtained at one pH. The efflux time, in seconds, of each run was converted into kinematic viscosity by consulting the calibration table (Table I made for the two viscometers).

In graphing, the kinematic viscosity values were plotted as the ordinates against the periods of digestion in minutes as the abscissas. This gave a smooth curve (Fig. 2) from which the time in minutes necessary for the proteolytic enzymes of malt to effect a 20% drop in kinematic viscosity was estimated. The point of intersection made by the horizontal dotted line (Fig. 2) with the curve for the digestion

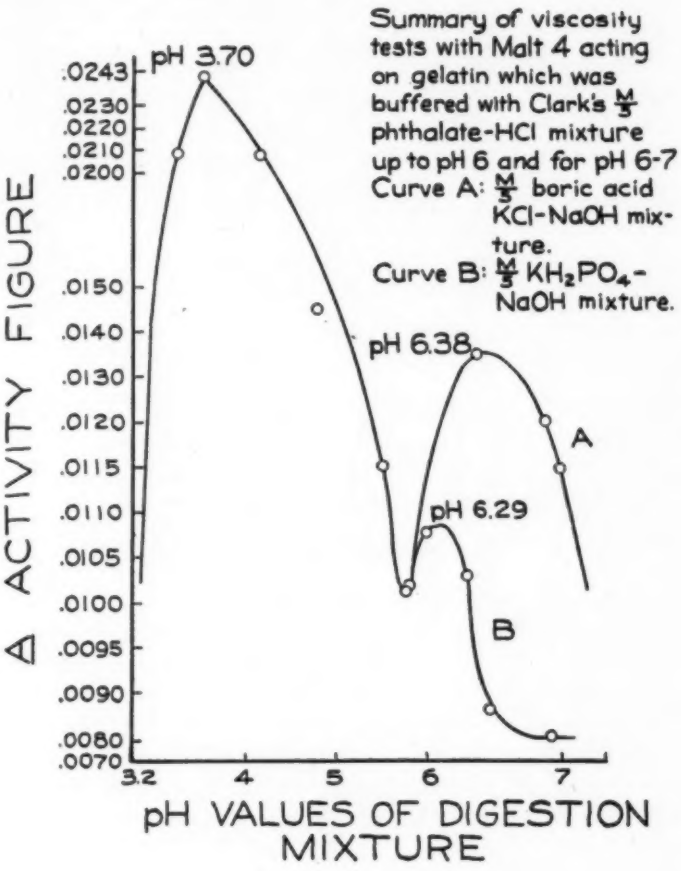


Fig. 6. Malt 4.

mixture denotes the time required to effect the 20% drop for the set pH. It is calculated by taking 80% of the viscosity of the blank. The summary graph for a series of curves (such as represented by Fig. 2) for one malt shows the rise and fall of activity as pH values of the digestion mixture are changed (Figs. 3 to 6). This graphic history of a malt operating at different pH values is obtained by plotting the pH values as abscissas and the ordinates as activity figures (*A*).

TABLE I

CONVERSION OF FLOW TIME TO KINEMATIC VISCOSITY

Viscometer A $K = At - B/t$; $A = 0.1140$; $B = 98.085$		Viscometer B $K = At - B/t$; $A = 0.1499$; $B = 133.37$	
Time in seconds	Kinematic viscosity	Time in seconds	Kinematic viscosity
40	21.08	40	26.61
41	22.82	41	28.93
42	24.53	42	31.20
43	26.21	43	33.44
44	27.87	44	35.64
45	29.51	45	37.82
46	31.12	46	39.96
47	32.72	47	42.08
60	52.06	60	67.72
61	53.47	61	69.57
62	54.86	62	71.42
63	56.26	63	72.26
64	57.65	64	75.10
65	59.01	65	76.92
66	60.38	66	78.73
67	61.75	67	80.53
68	63.10	68	82.32

Activity figures for each sample of malt were calculated from the formula, $A = 1/hg$ (Northrop, 1922), where A is the activity figure; h, the time in hours required to effect a 20% kinematic viscosity drop; and g, the concentration in grams per liter of ground malt.

Summary and Conclusions

The proteolytic activity of malt at different pH values can be measured viscometrically, thus locating an optimum pH.

A 20% drop in kinematic viscosity furnishes a desirable means of estimating proteolysis. It makes possible a determination at a given pH in two hours or less, thereby saving time and eliminating the disturbing factors that enter into prolonged digestion reactions.

A summary graph for each malt shows a wide range of pH values, indicating that the all-glass Koch viscometer is sensitive to even small changes in pH.

The amount of activity at an optimum point varies somewhat with the buffer used to produce the pH.

The use of 0.2M potassium acid phthalate buffer or of 0.2M HCl with gelatin produces the same effect. In either case there is a rise in activity with a decrease of pH—maximum occurring at pH 3.6 to 3.7.

Although only four malts are illustrated in this paper, actually ten malts were completely tested in the research. For all ten malts the maximum points found were between 3.6 and 3.7 for the one enzyme and between 6.3 and 6.5 for the other.

Acknowledgment

Appreciation is hereby extended to Mr. L. Ehrnst, Froedtert Grain and Malting Company, for all the practical assistance given this research and for the various samples of malt.

Literature Cited

Barr, Guy
1931 A monograph of viscometry. Oxford University Press, London.
Bingham, E. C., and Jackson, R. F.
1916 Standard substances for the calibration of viscometers. Scientific Papers of the Bureau of Standards No. 298, Aug. 8.
Britton, H. T.
1929 Hydrogen ions. Van Nostrand Pub. Co., New York.
Clark, W. M.
1923 The determination of hydrogen ions (2nd ed.). Williams and Wilkins Co., Baltimore, Md.
Ehrnst, L. W.
1938 Determination of proteolytic strength in malt. Modern Brewer **20**: 57.
Hagues, G.
1924 The effect of hydrogen ions in brewing processes. J. Inst. Brewing **30**: 298–304.
Hershel, Winslow H.
1917 Determination of absolute viscosity by short-tube viscometers. Technologic Papers of the Bureau of Standards No. 100, Nov. 9.
Hind, Lloyd H.
1938 Brewing science and practice, I. John Wiley and Sons, Inc., New York.
Hopkins, R. H., and Kelly, H. E.
1931 The proteolytic enzymes of green malt. Biochem. J. **25**: 256–259.
Hopkins, R. H., and Krause, C. B.
1937 Biochemistry applied to malting and brewing. Van Nostrand Pub. Co., New York.
Koch, J. R., Orthmann, A. C., and Sister M. Anselma Degenfelder, S. S. N. D.
1939 A digestion flask viscometer for measuring enzyme activity of bates using gelatin as a substrate. J. Am. Leather Chem. Assoc. **34**: 489–514.
Koch, J. R., Nelson, O., and Ehrnst, L.
1939 Viscometer for routine determination of proteolytic activity of malts. Ind. Eng. Chem. (Anal. Ed.) **11**: 35–41.
Kolbach, P., and Simon, H.
1936a Über die proteolytische Kraft von Malzauazugen und Wuerzen. Wochenschr. Brau. **53**: 297–299.
1936b Proteolytic power of malt infusions and worts. The Brewers Technical Rev. **11**: 351–353.
Laufer, Stephen
1937 Studies on the proteolytic activity of barley malt. J. Assoc. Offic. Agr. Chemists **20**: 307–330.
1938 Reports on proteolytic activity of malt. J. Assoc. Offic. Agr. Chemists **21**: 160–164.
Lüers, H., and Malsch, Ludwig
1929 Zur Kenntnis der proteolytischen Enzyme des Malzes. Wochenschr. Brau. **46**: 265–269, 275–280.
Lundin, H.
1923 Ein Beitrag zur Kenntnis der proteolytischen Enzyme des Malzes. Biochem. Z. **131**: 193–218.
Mill, C. K., and Linderstrøm-Lang, K.
1927–29 Note on the proteolytic enzymes in green malt. Compt. rend. trav. lab. Carlsberg **17**: 1–14.
Northrop, J. H.
1922 The mechanism of the influence of acids and alkalies on the digestion of proteins by pepsin and trypsin. J. Gen. Physiol. **5**: 263–274.
1923 A method for the quantitative determination of trypsin and pepsin. J. Gen. Physiol. **5**: 353–358.
Sheely, M. L.
1923 The standardization of commercial viscometers. Ind. Eng. Chem. **15**: 1109–1114.
1932 Glycerol viscosity tables. Ind. Eng. Chem. **24**: 1060–1064.

Tauber, Henry
1937 Enzyme chemistry. John Wiley and Sons, New York.
Van Laer, M. M.
1923 The possibilities of increasing the brewer's extract. J. Inst. Brewing **29**: 202–218.
Waksman, S. A.
1926 Enzymes. Williams and Wilkins Company, Baltimore, Md.
Wallerstein, Leo
1939 Enzyme preparations derived from microorganisms. Wallerstein Lab. Comm. Sci. Pract. Brewing **7**: 5–19.

THE DISTRIBUTION OF PHYTIC ACID IN WHEAT AND A PRELIMINARY STUDY OF SOME OF THE CALCIUM SALTS OF THIS ACID

J. G. Hay

Joseph Rank, Ltd., Reigate, Surrey, England

(Received for publication October 6, 1941)

The phytic acid phosphorus in the pericarp of the wheat grain is to a large extent fixed in the form of calcium magnesium phytate, with the result that in bran between 90% and 100% of the total phosphorus is phytic acid combined as a calcium magnesium salt of the hexaphosphoric ester of inositol. This water-insoluble salt no doubt secures the phosphorus from loss through diffusion when the grain is exposed to rain, but liberates it in an available inorganic form by action of phytase when the germinating plant requires it.

In present commercial wheat mill products the phytic acid phosphorus is directly proportional to the fiber content. With white flour, 85% wheat meal, 100% whole meal, fine wheat feed, and bran, the graph obtained when the fiber content is plotted against the phytic acid phosphorus is a straight line, as shown in Table I.

TABLE I

Fiber and Phytic Acid Phosphorus

	Fiber	Phytic acid phosphorus
	%	%
75% extraction white flour	0.28	0.025
85% extraction wheat meal	0.95	0.122
100% extraction whole meal	1.96	0.203
Fine wheat feed	6.10	0.660
Bran	9.75	1.105

The product referred to as fine wheat feed is the tails (from all the machines) which pass through a certain mesh, but without any additions such as ground shriveled wheat, which may be added to commercial "weatings" or sharps.

The figures given are typical of those obtained in an ordinary commercial milling of mixed wheats, with red wheats in great preponderance, as is usually the case. There is some evidence to indicate that certain white wheats may have a lower phytic acid content than red ones, as indicated in Table II.

TABLE II
PHYTIC ACID CONTENT OF RED AND WHITE WHEATS

	Fiber in wheat	Phytic acid phosphorus in wheat	Fiber in bran	Phytic acid phosphorus in bran
	%	%	%	%
White Victorian	1.95	0.17	10.58	0.78
White Pacific	2.1	0.175	10.18	0.72
Red No. 1 Manitoba	2.05	0.22	9.65	1.07

This point, if proved, is of interest, as it may account for the hitherto unexplained fact that farmers are prepared to pay a premium for white bran over red, although on analysis red bran averages a higher protein content than white, the other constituents being about the same: so possibly stock producers have found by experience that white bran gives better results in feeding stock than red, which may be due to its lower phytic acid and consequently lower calcium-immobilizing action on other foods.

One sample of white English wheat examined did not differ materially from red wheats in phytic acid phosphorus content, but before the War, when it was permissible to make "divides" in milling, by far the greater part of white bran was derived from white wheat other than English—such as Australian and White Pacific—so its influence on white bran would be small.

There are certain exceptions in the individual tails from the various centrifugals (which collectively form the fine wheat feed) in their phytic acid phosphorus content in relation to fiber content; but it must be borne in mind that the crude fiber content as determined is not a true content of cellulose, being the amount of material insoluble under certain conditions in specified strengths of acid and alkali, so that when the softer and probably more soluble tissues of the germ and the parenchymatous cells of the endosperm are compared in fiber content with those tougher structures in the pericarp, the comparison is not quite the same. However, in a product such as pure germ, as free as possible from adhering bran particles, the phytic acid phosphorus is much higher in relation to fiber content than in the case of bran, as shown in Table III.

The tails from machines containing germy stock, such as J. scalper tails, do not show a great discrepancy from the theoretical point on the

TABLE III

PHYTIC ACID PHOSPHORUS IN GERM AND BRAN

	Fiber	Phytic acid phosphorus	Ash	Total phosphorus
	%	%	%	%
1. Germ	2.15	0.52	4.41	1.12
2. Germ	2.05	0.49	4.53	0.94
Bran, same milling as germ No. 2	10.3	1.17	5.54	1.21

graph for their fiber content, for in this case the fiber content is relatively high, due to bran particles, a typical sample giving 5.35% fiber and 0.60% phytic acid phosphorus.

Another exception is found in such stocks as M. centrifugal tails. This product is a light-colored, fluffy material, containing very few bran particles; it is only a small percentage of the total fine wheat feed. Under the microscope it is found to contain a considerable number of parenchymatous cells from the interior of the endosperm. Figures for this product are shown in Table IV.

TABLE IV

DATA FOR M. TAILS

	Fiber	Phytic acid phosphorus	Ash	Total phosphorus
	%	%	%	%
M. tails	2.15	0.46	2.70	0.54

To investigate the point as to whether there was a high percentage of phytic acid in the endosperm cellular tissue, 7 pounds of purest endosperm obtainable on the mill, in the form of semolina free from bran particles, was rolled between smooth rolls on an experimental mill, the tails from a No. 11 silk were rerolled, and the tails from a similar silk, amounting to 0.23% of the semolina, were examined under the microscope and found to contain a large proportion of parenchymatous cells, as shown in Table V.

TABLE V

DATA ON ENDOSPERM

	Fiber	Phytic acid phosphorus	Total phosphorus	Calcium	Ash	Percentage of phosphorus as phytic acid phosphorus
	%	%	%	%	%	%
Tails from pure endosperm	1.15	0.435	0.437	0.076	2.04	99.5
Flour from pure endosperm	0.12	0.025	0.079	0.019	0.38	31.6

These results indicate that the small amount of phytic acid phosphorus in high-grade flour is derived from the small particles of white endosperm intercellular tissue, which go through the silk-clothed dressing machines, and not from bran particles which are almost entirely absent in a well milled high-grade flour.

The tables below give the figures for various wheat mill products: Table VI for a mill working on the Simon's system, Table VII on

TABLE VI

ANALYSIS OF PRODUCTS FROM MILLING BY SYSTEM A

Product	Fiber	Phytic acid phosphorus	Total phosphorus	Percentage of total phosphorus as phytic acid phosphorus	Ash	Calcium
	%	%	%	%	%	%
Mill feed (100% whole meal)	1.76	0.203	0.35	57	1.56	0.046
72% extraction white flour	0.18	0.023	0.097	24	0.46	0.020
85% extraction wheat meal	0.91	0.11	0.189	59	0.90	0.041
Fine wheat feed	6.15	0.662	0.832	79.5	3.78	0.085
Bran	9.45	1.07	1.18	90	5.46	0.094
Germ	2.20	0.52	1.12	46	4.41	0.053
J. scalper tails	5.35	0.60	0.86	70	3.60	0.069
M. tails	2.15	0.46	0.54	85	2.70	0.037

TABLE VII

ANALYSIS OF PRODUCTS FROM MILLING BY SYSTEM B

Product	Fiber	Phytic acid phosphorus	Total phosphorus	Percentage of total phosphorus as phytic acid phosphorus	Ash	Calcium
	%	%	%	%	%	%
Mill feed (100% whole meal)	2.10	0.215	0.334	64	1.52	0.057
75% extraction white flour	0.25	0.053	0.117	45	0.53	0.025
85% extraction wheat meal	1.0	0.135	0.204	66	0.97	0.042
95% extraction wheat meal	1.62	0.162	0.271	60	1.29	0.055
Fine wheat feed	6.80	0.73	0.86	85	3.96	0.084
Bran	10.30	1.17	1.21	97	5.54	0.096
Germ	2.05	0.49	0.94	52	4.53	0.051
M. tails (equivalent)	3.55	0.715	0.742	96	3.39	0.080

another milling system. Both mill grists were similar, containing 65% Manitoban wheats, 15% Plate, 10% Australian, and 10% various wheats. The phytic acid phosphorus is in all cases estimated on the raw products; after fermentation and cooking in bread or similar products, varying degrees of hydrolysis (by phytase and other means) will take place, giving some inositol and inorganic phosphorus, varying with temperature, time of fermentation, and other factors.

The flour and tails from the various machines not represented in the tables all gave figures which, within the limits of experimental error, fall on the line of the graph shown in Figure 1. The figures show the

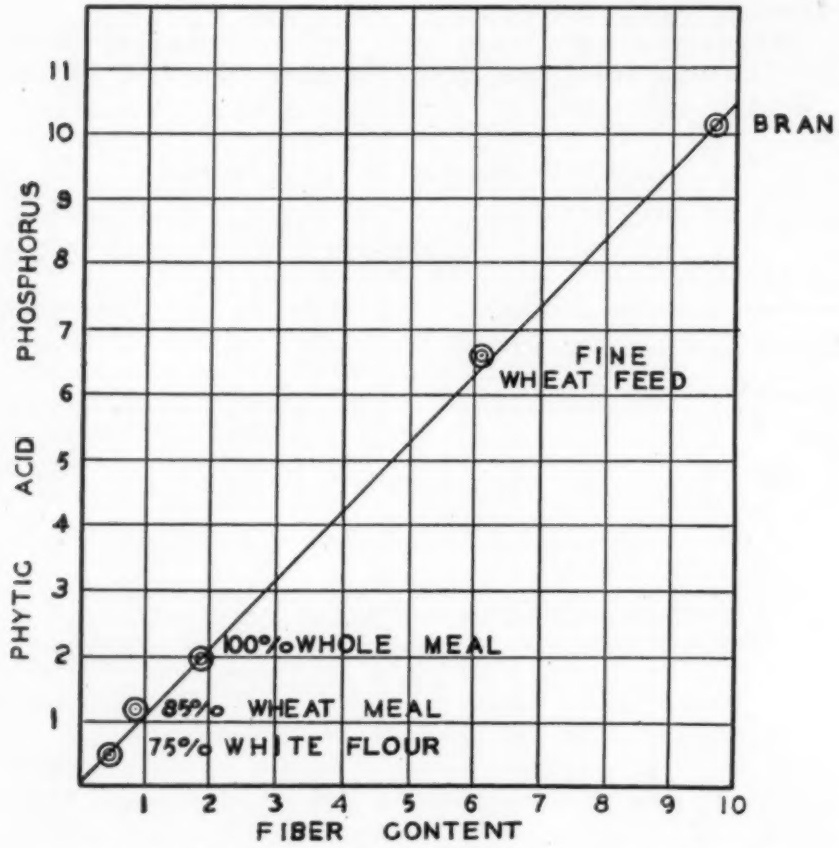


Fig. 1. Phytic acid phosphorus in relation to fiber content and percentage of extraction.

rapid increase in phytic acid phosphorus with the degree of extraction of the flour; thus 75% extraction flour has over double the amount of 72% flour. The lowest figure obtained was in C reduction flour, which gave 0.011% phytic acid phosphorus, which is about one-hundredth of the amount found in bran; this flour in pre-war days would have been classed as patent or high grade.

A preliminary investigation was made on the calcium salts of phytic acid. It was found that in the phytic acid extracted from bran, by the method of Harrison and Mellanby (1939) for oatmeal, the calcium salt obtained at pH values likely to exist in the digestive system

was a hexa-calcium salt, with a ratio of calcium to phosphorus of 40:31, but, as pointed out by the authors referred to, the phytic acid, in immobilizing a part of the calcium, also combines with varying amounts of magnesium, forming double salts. Accordingly no useful figures on the amount of calcium immobilized can very well be obtained by a theoretical study of the amount of phytic acid and calcium present in a product, as it is complicated by the amount of magnesium with which they will combine, and this, again, may depend upon various factors. The compositions of the calcium salts, produced at different pH values and dried at varying temperatures, are shown in Table VIII.

TABLE VIII

DATA ON CALCIUM SALTS

Solution in	pH	Temperature of drying	Calcium	Phosphorus	Probable composition
		deg C	%	%	
Dilute HCl	2.5	110	13.8	22.1	$Ca_3P_6\cdots$
50% acetic acid	1.2	20	15.3	17.9	$Ca_4P_6\cdots$
50% acetic acid	1.2	100	15.7	18.5	$Ca_4P_6\cdots$
50% acetic acid	1.2	110	16.5	19.1	$Ca_4P_6\cdots$
50% acetic acid	1.2	125	17.0	19.4	$Ca_4P_6\cdots$
50% acetic acid	1.2	150	18.2	21.3	$Ca_4P_6\cdots$
Acetic acid	3	110	19.8	18.8	$Ca_5P_6\cdots$?
Acetic acid	4.5	110	23.3	19.2	Mixture of $Ca_4P_6\cdots$ and $Ca_6P_6\cdots$?
Acetic acid	6.0	100	23.8	18.7	$Ca_6P_6\cdots$
Acetate	8.0	150	25.4	19.6	$Ca_6P_6\cdots$
Acetate	10.0	100	23.4	18.2	$Ca_6P_6\cdots$
Acetate	10.0	120	25.4	19.65	$Ca_6P_6\cdots$

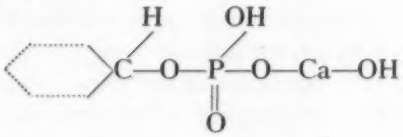
The tricalcium salt is very soluble in water; it is hygroscopic, but only slightly soluble in 90% alcohol. On boiling with water, or heating at 110°C., it appears to decompose into a higher calcium salt and phytic acid. The 1% cold water solution has a pH of 3.8.

The tetracalcium salt is produced in 50% acetic acid in which it is only slightly soluble; it is soluble in dilute acetic acid and water, but, like the tricalcium salt, gives an insoluble higher salt and phytic acid on boiling or heating dry.

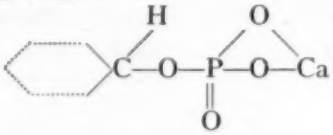
The pentacalcium salt could not be definitely isolated, but at pH3 the calcium salt precipitated agrees with this composition, although it may be a mixture of the tetra- and hexa-salts.

On approaching neutrality at pH 5.8 and on the alkaline side up to pH10, the hexacalcium salt is produced. This salt appears to be basic in character and its composition may be $C_6H_{18}O_{30}P_6Ca_6$ when dried at 100°C, but when heated to 150°C it loses three molecules of water, giving $C_6H_{12}O_{27}P_6Ca_6$, so it is possible that each of the six P atoms is

combined as below:



and when heated to higher temperatures than 100° loses one to six molecules of water, giving:



although other alternative formulae could explain this dehydration.

Experimental

The phytic acid phosphorus was estimated by the method detailed by McCance and Widdowson (1935), based on Brigg's original method. In making the first extraction with 0.5*N* HCl, the volume was made up to 100 ml. Thus, if 10 g of fine wheat feed was to be extracted, 91.5 ml of 0.5*N* HCl was added, giving a total volume of 100 ml of wheat-feed suspension. After two hours of shaking, 5 ml of the filtrate was used in the determination. This was proved to be nearly correct by removing 75 ml from the 100 ml and making up to 100 ml again with fresh 0.5*N* HCl, and extracting again for two hours. Then by determining the phytic acid in 20 ml of the filtrate instead of 5 ml as in the first extraction, almost identical figures were obtained, showing that the phytic acid is distributed evenly throughout the permeable mass, in the same proportion as in the surrounding liquid.

The second extraction had a tendency with some products to give a slightly higher result than the first—about 3% or 4%—indicating that the first extraction is not quite complete; thus a sample of bran, giving 1.17% phytic acid phosphorus on second extraction, gave 1.21%/4 phytic acid phosphorus; but differences such as this are within the experimental error of the method.

The total phosphorus and calcium were estimated in the usual manner, the calcium precipitated from dilute acetic acid as oxalate, titrated with 0.05*N* $KMnO_4$, and the phosphate after separation as phospho-molybdate precipitated and weighed as $Mg(NH_4)PO_46H_2O$ on cintered glass Gooch, as described by Fales (1928).

Fiber was determined by the official method described in the Fertilisers and Feeding Stuffs Regulations (1928). In this connection it should be mentioned that flour and similar products with a fiber con-

tent below 0.5% give unsatisfactory results, as duplicate analyses may give nearly as wide variations as comparisons with a sample of bran containing 20 or more times as much fiber.

The preparation of a solution of sodium phytate from bran was made as described by Harrison and Mellanby (1939), for extraction from oatmeal: the solution was made slightly acid with acetic acid and boiled for two minutes to remove CO_2, then rapidly cooled, and the pH adjusted by further addition of acetic acid or CO_2-free NaOH. The 50% acetic acid solution contained $CaCl_2$, and the precipitate was washed with 50% acetic acid till free from Cl. The tricalcium salt was precipitated from the dilute HCl solution by pouring this solution into nine or ten times its volume of 95% alcohol and washing the precipitate with 95% alcohol until free from Cl.

The hexacalcium salt was formed by adding calcium chloride or calcium acetate solution to the sodium phytate solution which had been boiled with dilute acetic acid for two minutes to remove CO_2, before adding pure NaOH to the required pH. There was no evidence that a higher calcium salt than the hexa-salt could be produced even in a strongly alkaline solution, under these conditions; the precipitate formed contained slightly more calcium than the theoretical for the hexa-salt, but this was probably due to a small quantity of tricalcium phosphate resulting from a slight hydrolysis of phytic acid by boiling with dilute acetic acid.

The calcium and phosphorus were estimated in these salts by oxidation in a hard glass test tube with H_2SO_4 and $HClO_4$ as described by Harrison and Mellanby; the calcium was precipitated in dilute acetic acid as oxalate and phosphorus directly on the filtrate as $Mg(NH_4)PO_46H_2O$.

Conclusions

In wheat mill commercial products the phytic acid phosphorus is proportional to the fiber content; but in certain constituents of those products, notably germ and parenchymatous cellular tissue of the endosperm, the phytic acid phosphorus is in a higher ratio to the fiber than in the pericarp tissue.

The calcium phytate likely to be produced in the alimentary tract is the hexacalcium salt.

Literature Cited

Fales, H. A.
1928 Inorganic chemical analysis. Appleton-Century, New York.
Harrison, D. C., and Mellanby, E.
1939 Phytic acid and the rickets-producing action of cereals. Biochem. J. **33**: 1660–1680.
McCance, R. A., and Widdowson, E. M.
1935 Phytin in human nutrition. Biochem. J. **29**: 2694–2699.

THE ACTION OF AN OXIDIZING AGENT IN BREAD DOUGH MADE FROM PATENT FLOURS

J. C. Baker, H. K. Parker, and M. D. Mize

Wallace & Tiernan Laboratories, Newark, New Jersey

(Read at the Annual Meeting, May 1941)

The action of oxidizing agents in altering the baking properties of dough has been the subject of intensive investigation by many workers. These investigations have been recently reviewed by Sullivan, Howe, Schmalz, and Astleford (1940). In their conclusions they suggest that the oxidation reaction causing baking improvement occurs in the protein of the flour but were unable to identify its character. Other workers, such as Jørgensen (1936) and Balls and Hale (1936), have stated that the improvement is due to the effect of the oxidizing agent on proteolytic enzymes or their activators in the flour, whereby proteolytic action is inhibited. However, Hale (1939) and Landis (1940) were able to find so little proteolytic enzyme in patent flours that much doubt is thrown on this hypothesis. The experiments of Read and Haas (1937) show that bromate used at commercial rates of application and commercial pH range did not inhibit proteases.

Bread doughs often soften with time. This softening is perhaps the reason many workers believe that proteolytic enzymes are involved in the reactions that affect bread quality. In order to study this softening of doughs we have modified the technique of Halton and Scott Blair (1937). Instead of extruding doughs and trying to obtain a test piece of fixed diameter, we have worked on a test piece of a definite weight and molded it to a definite length, letting it take whatever diameter was needed to accommodate the gases in the dough. The fixed weight of dough was always molded to the same length and placed on a mercury bath. Two ink marks were placed at a fixed distance apart on the dough surface, leaving sufficient dough outside of the marks for grasping it at each end. One end of the dough was attached to a spring scale while the other end was gripped by the fingers, stretched to a definite tension, and held for a definite length of time; then quickly released. Two readings were thus obtained—the total extension and the spring-back (elastic extension). Flow was calculated by recording the difference between the two readings. The spring-back and flow were recorded in all of our work. The sum of the two figures gave the total extension and the ratio of the two values could also be calculated.

It was noted that spring and flow change together in the same direction, but the changes of greatest magnitude were found in flow. In order to simplify a study of our results, flow has been the only

variable considered here. This is considered justifiable, as flow is largely responsible for the changes in ratio of viscosity to elasticity in doughs and certainly results in coalescence and breakage of dough bubbles in bread making.

In order to test the effects of oxidation and yeast upon bread dough, two series of doughs were made, one with and the other without yeast, —each series included doughs with moderate and heavy degrees of oxidation,[1] respectively. Table I shows a complete summary of the results obtained in these series of tests. In Figure 1 the results of flow have been charted.

TABLE I

EFFECT OF OXIDATION ON THE ELASTIC SPRING (S) AND VISCOUS FLOW (F) OF DOUGHS, WITH AND WITHOUT YEAST

Doughs	Time after mixing					
	At once		30 min		90 min	
	S mm	F mm	S mm	F mm	S mm	F mm
YEASTLESS DOUGHS						
Unoxidized	33	18	44	23	70	41
" + 5 ppm $NaClO_2$	33	16	38	15	48	24
" + 40 ppm $NaClO_2$	36	18	31	6	28	4
DOUGHS + 2½% YEAST						
Unoxidized	34	20	32	12	34	18
" + 5 ppm $NaClO_2$	31	17	28	13	25	7
" + 40 ppm $NaClO_2$	39	14	24	5	16	2

All doughs irrespective of oxidation or presence of yeast showed substantially the same amount of flow when taken immediately from the mixer. The yeastless, unoxidized dough showed an increase in the amount of flow of the dough on standing, suggesting that some proteolytic action was taking place. The flow was largely prevented in the dough by a moderate degree of oxidation. This dough remained nearly unchanged with time, suggesting that such oxidation eliminated the proteolytic effects. A high degree of oxidation of the yeastless dough caused tightening of the dough with a marked decrease in flow, which progressively became less as time went on until the dough had very little flow at the end of the test period. This change in flow,

[1] Baker and Mize (1941) describe advantages of sodium chlorite as an oxidizing agent for research work.

opposite to that of the unoxidized dough, was not suggestive of a proteolytic effect.

Further, the proteolytic theory was not supported when yeasts were added to the doughs, for then the softening effects in the dough were almost entirely eliminated and the tightening effects produced by oxidation were intensified in every case. Apparently the yeast produced an effect very similar to that of the oxidizing agent. This suggested that both were reacting upon or affecting the same ingredient in the dough. One of the purposes of this work was to search for and investigate the nature and behavior of this ingredient.

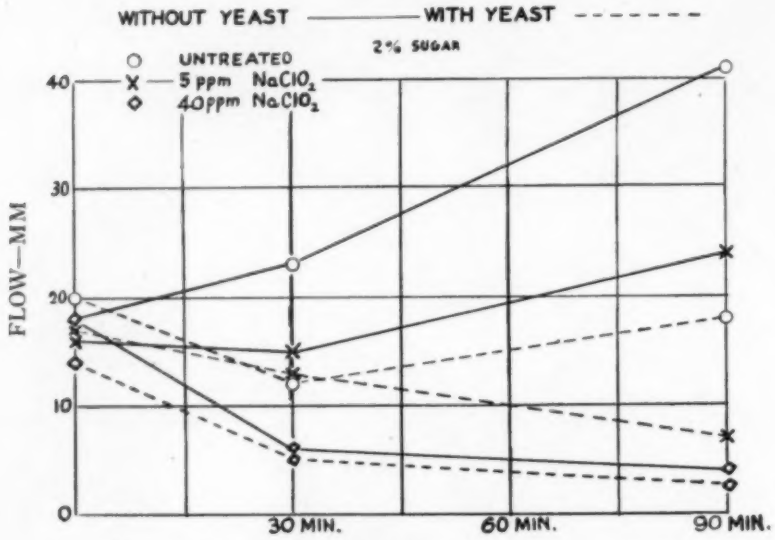


Fig. 1. Effect of oxidation on viscous flow of doughs.

Bread was made from flour thoroughly extracted with petroleum ether and carbon tetrachloride. The "no-time" process of baking was used on these flours because the effects of oxidation are always more pronounced in this method of baking. In Figure 2, which shows the resulting bread, the first loaf was unoxidized. The second was made from a portion of the same dough to which 35 ppm of sodium chlorite was added. The very marked improvement in the baking qualities was evidence that the extraction of flour did not remove from the flour that property which resulted in baking improvement when the doughs were oxidized. Hence it appeared that this property is not due in any marked degree to the material extracted from flour by the fat solvents.[2] In order to determine in which portion of flour the reacting material

[2] Kosmin (1934) has shown interesting response to oxidizing agents in glutens from extracted flours.

may be found, dough was separated by a gluten washing process into gluten, starch, "amylodextrin" (by centrifuging),[3] and gluten wash solution. Each fraction was made up to one liter in ¾% salt solution, and the gluten fraction was dispersed in this volume of solution with a

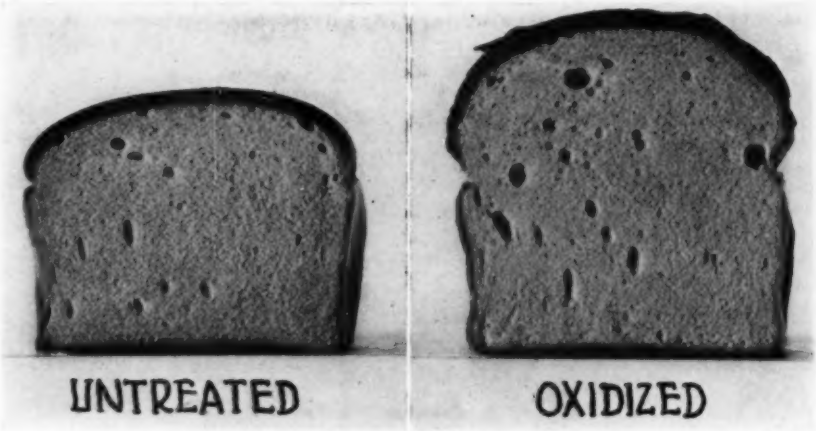


Fig. 2. The response of extracted flour to oxidation.

Waring mixer. To each solution the same amount of sodium chlorite was added and the solutions were permitted to stand 24 hours. The amount of sodium chlorite remaining was determined by titration. Table II gives the results of this experiment.

TABLE II

REACTION OF FLOUR CONSTITUENTS TO OXIDATION
(Each fraction from 100 g of flour reacted for 24 hours in 1 liter of ¾% NaCl solution with 9 mg $NaClO_2$)

Constituent	$NaClO_2$ remaining after 24 hours
	mg
Gluten	0.0
Starch	8.6
Amylodextrin	8.7
Solubles (gluten wash solution)	0.0
Blank—1 liter ¾% NaCl solution	9.0

The fractions containing gluten and gluten wash solution consumed all the sodium chlorite added. The fractions containing starch and amylodextrin consumed substantially none of the sodium chlorite in 24 hours, indicating clearly that the reactive material is to be found either in the gluten itself or in the gluten wash solution. We did not further investigate the relation of starch and "amylodextrin" to this

[3] This material was described by Sandstedt, Jolitz and Blish (1939), page 781.

problem. The findings with reference to the lipoid and starch portions of flour are in agreement with results reported by Sullivan, Howe, Schmalz, and Astleford (1940).

Preliminary observations of the action of oxidizing agents upon gluten showed very marked changes in gluten properties. An apparatus was therefore built to study gluten physical properties.

Fig. 3. Gluten testing device.

Figure 3 shows the device, which consists of a cylindrical wire cage attached to a tube in which the operation of a plunger is actuated by air pressure. The gluten is placed so that it fills the cage and extends into the cylinder. The plunger pushing down forces it to fill the space and bulge out through the large meshes in the wire screen. Upon release of the pressure, the gluten springs back to relieve the strain. The downward and upward motions of the plunger are measured and the difference between the two measurements indicates the flow or viscosity of the gluten. The value of this device lies in the fact that the gluten is confined to a definite shape, which makes it possible to secure reliable and reproducible measurements. Table III shows the physical properties of a series of glutens prepared from one flour under different

conditions. The results are expressed in units of the plunger scale as "spring" and "flow." There is no numerical relationship between these readings on gluten and those above on dough as the two instruments involved give results which cannot be translated from one to the other.

TABLE III

EFFECT OF DOUGH ENVIRONMENT ON ELASTIC SPRING (S) AND PLASTIC FLOW (F) OF GLUTEN

Origin of gluten	S	F
1. Yeastless dough washed in 3 liters of ¾% NaCl solution after mixing	1.76	0.64
2. Gluten No. 1 allowed to stand 4 hours	1.80	0.55
3. Dough of No. 1 stood 4 hours before washing	1.57	0.57
4. Same dough containing 2½% yeast stood 4 hours before washing	1.18	0.16
5. Gluten No. 1 washed in 3 additional 3-liter solutions	1.26	0.16
6. Gluten No. 5 worked in 40 ppm $NaClO_2$ in last solution	1.17	0.14
7. Gluten No. 1 worked in 200 ml of ¾% salt solution +40 ppm $NaClO_2$	1.11	0.22
8. Gluten No. 1 worked in 200 ml of ¾% salt solution only	1.34	0.51
9. Gluten No. 1 worked in 200 ml of ¾% salt solution containing 200 ppm papain and then stood one hour	1.70	1.12

Note: Gluten No. 1 washed 20 minutes in 3-liter solution, then two additional 200 ml 5-minute washes (all other washings above were 20 minutes each).

In the gluten experiments reported in this paper all operations have been conducted in ¾% salt solution prepared with boiled distilled water saturated with carbon dioxide. A current of carbon dioxide was also bubbled through the solution during the experiments. All doughs had been mixed in the absence of air in a carbon dioxide atmosphere, unless otherwise stated. Throughout this paper one flour only has been used—a Southwestern patent containing 11.25% protein and 0.40% ash.

It is to be noted in Table III that the changes in spring are proportionally smaller than are the changes in flow. In order to simplify the consideration of the effects produced on the gluten, flow only will be discussed.

Gluten No. 1 was the basic gluten with which all other glutens considered in Table III were compared. It had the greatest flow of any of the glutens. *Gluten No. 2*, after standing four hours, showed a small amount of tightening and loss of flow. This is in contrast to the softening sometimes observed in doughs. *Gluten No. 3* showed that if the dough itself stood four hours before washing the result was substantially the same as in the instance where the washed gluten stood four hours. If the theory that proteolytic enzymes soften gluten in a dough is correct, it is hardly conceivable that this should be the case, for the washing of a gluten must remove much of the enzymatic material.

Gluten No. 4 was similar to No. 3 with the exception that 2½% yeast acted on the dough for the four hours. The flow properties of the gluten had now almost entirely disappeared because of the fermentation. This indicates that the changes produced in dough by yeast, as shown in Table I, are found in the gluten. Tightening of the dough was accompanied by corresponding tightening of the gluten, indicating that changes in dough properties caused by yeast are also to be found in the gluten itself. *Gluten No. 5* was a portion of No. 1 washed in three additional three-liter portions of solution. It may be noted that this gluten, merely by washing, exhibited the same properties as were found in the gluten from the dough which had been acted upon by yeast. This indicates that the material whose removal is responsible for the tightening of the gluten is soluble in the solution and that by washing it out a reaction similar to that with yeast is obtained.

Gluten No. 6 was prepared exactly like No. 5 except that the last three-liter portion of wash water contained 40 ppm of sodium chlorite. The manipulation of this gluten in the oxidizing solution has produced only a very slight tightening. *Gluten No. 7* was produced by subjectnig another portion of No. 1 to a similar treatment in a small volume of wash water containing 40 ppm of sodium chlorite. It is to be noted that the soft gluten now became a very tight, compact, rubbery mass, similar to the glutens obtained either by yeast or by extreme washing. In order to show that this effect was not due to the small amount of water in which the gluten was manipulated, *gluten No. 8* was a similar gluten worked in 200 ml of solution containing no sodium chlorite. Here only a slight tightening of the gluten occurred. Finally, gluten No. 1 was washed in a similar 200-ml portion of solution containing 200 ppm of commercial papain, as shown by *gluten No. 9*. This gluten exhibited a marked softening from the treatment, indicating that proteolytic enzymes, when present, soften gluten, giving a change in properties opposite to that obtained from all of the above treatments. This indicated that the material in gluten causing its response to oxidizing agents is not proteolytic in nature and suggested a further study of the water solubles.

In an attempt to further extract the solubles from gluten, it was dispersed in a "Waring Blendor" [4] and the dispersed gluten suspension poured into centrifuge tubes and whirled. The gluten came together either at the top or at the bottom of the tube, or both, and was collected as firm, packed gluten, appearing substantially the same as before it went into the blender. However, this gluten was unusually tough and, though extensible, it showed the least flow of any gluten prepared.

[4] Freilich (1941) described this apparatus and its applicability to flour suspension problems. Our modification maintains the material under CO_2 during preparations.

This dispersed gluten was then redispersed and collected, and the same operation repeated a total of six times. The resulting gluten was similar to that obtained from the first dispersion. The successive solutions in which this gluten was dispersed were analyzed for nitrogen, as shown in Table IV.

TABLE IV

SOLUBLE PROTEIN FROM SUCCESSIVE WASHINGS OF GLUTEN BY HAND AND BY MECHANICAL DISPERSION

(270 g flour washed in oxygen-free CO_2-saturated ¾% salt solution)

Wash solution analyzed	Protein in total wash solution		Concentration of protein in wash solution
			g per l
First 1.8 liter wash solution	4.457	1800 ml	2.48
After three more 1.8-liter 20-minute workings	0.235		0.13
After dispersion in 375 ml of solution	0.582	375 ml	1.55
After 2nd dispersion	0.354		0.94
After 3rd dispersion	0.268		0.72
After 4th dispersion	0.222		0.59
After 5th dispersion	0.184		0.49
After 6th dispersion	0.178		0.48

The four gluten wash solutions each amounted to 1800 ml. The dispersions were each in 375 ml of solution and were made with 90 grams of wet gluten.

It is to be noted that there is a large increase in the solubility of the protein upon dispersion. By breaking up the gluten into an extremely fine suspension, as obtained in a Waring mixer, a large amount of protein was obtained which was so soluble that it could not be centrifuged or filtered out. On successive dispersions of the remaining gluten, decreasing amounts of soluble nitrogenous material were found until on the fifth and sixth dispersions nearly a constant solubility was found, suggesting that the soluble protein was not largely a component of gluten itself. The first dispersate protein fraction exhibits the characteristic properties of a proteose. It was very slowly coagulated with heat and required saturation with ammonium sulfate to obtain complete precipitation. Thus it seems that this protein is different from the gluten itself. The presence of this dispersible fraction in gluten, which can be removed by very violent mechanical means from the body of the gluten, suggests that this is a material which may be responsible for the slippage in the gluten before dispersion.

In order to study further the dispersible fraction of gluten, certain glutens reported in Table III were dispersed and the amount of the dispersible protein measured in the first dispersate liquor as shown in Table V.

TABLE V

PROTEINS SOLUBLE BY DISPERSING GLUTENS IN WARING BLENDOR

(Soluble protein in grams per liter)

Preparation of gluten	Gluten wash solution	First dispersate liquor from gluten obtained by	
		Ordinary washing	Extreme washing
Gluten from unfermented dough	2.32	1.78	1.55
Gluten from 4-hour fermented dough	2.24	1.45	1.15
Gluten from 4-hour unfermented dough	2.39	1.49	1.36

Note that ordinary gluten carries more dispersible nitrogenous material than gluten obtained by thorough washing or by fermentation, suggesting that these two operations have each removed some of the dispersible fraction and thus rendered the glutens less likely to flow when tested as shown in Table III.

In order to find the response of gluten to the solubles in flour, a concentrated gluten wash water was prepared. Gluten was washed from a dough in an equal weight of ¾% salt solution. The starch and "amylodextrin" were centrifuged from the wash solution, which was then reused to continue the washing of the gluten, thereby obtaining substantially complete removal of starch and "amylodextrin." Gluten prepared in this manner exhibited unusual properties, some of which are shown in Table VI.

TABLE VI

GLUTEN WASHED FROM A DOUGH IN AN EQUAL WEIGHT OF ¾% SALT SOLUTION AND REWASHED IN SAME SOLUTION CENTRIFUGED TO REMOVE STARCH AND AMYLODEXTRIN

	Spring	Flow
Gluten as prepared	3.13	2.13
Same gluten washed in an equal volume of ¾% salt solution	1.26	0.44
Same gluten worked in original centrifuged wash solution +0.1% $NaClO_2$	1.87	0.21

It exhibited approximately three times the flow of any of the glutens previously prepared. However, when washed in one small equal volume of salt solution alone, its flow dropped enormously, giving a gluten with less flow than one prepared by the ordinary method. When this gluten was similarly washed in water containing sodium chlorite, it tightened and nearly all of its flow properties disappeared. Apparently one can dissolve out the property that is responsible for flow in gluten or one can oxidize it so that its effects disappear. These effects can be produced and measured in so short a time interval that interpretation of results by enzymatic explanation seems improbable.

The remarkable change in gluten properties produced by washing in a small volume of salt solution led us to study the properties of a gluten which was merely manipulated in a smaller volume of solution for increasing lengths of time. It then became apparent that merely working a gluten in a solution without changing the water at all, causes it to tighten almost as much as does extreme washing, yeast, or oxidation. It is now apparent that gluten is composed of materials which can be entangled or enmeshed by mechanical working so that the molecules do not slip by one another. This suggests that gluten may be composed of molecules that are shaped like coiled springs. If one would take a mass of small coil springs and work them together, changes in the properties of the mass similar to that observed in gluten would be obtained. The more they are worked the harder they are to separate. The dispersible fraction which is in gluten may interfere with the intermeshing of the coils and may also lubricate the molecules so they readily slip apart. In order to study this hypothesis a series of glutens were worked in the strong gluten wash solutions. The results are reported in Table VII.

TABLE VII

EFFECT OF WORKING GLUTENS IN CONCENTRATED CENTRIFUGED GLUTEN WASH SOLUTIONS

(S = spring; F = flow)

Type of gluten	Unworked		Worked	
	S	F	S	F
Gluten by 5 dispersions	1.17	0.13	1.16	0.09
Gluten by thorough washing	1.36	0.18	1.23	0.29
Gluten from fermented dough	1.27	0.15	1.15	0.14
Gluten by ordinary washing	1.46	0.54	1.53	0.70

The redispersed gluten which was substantially free from dispersible material gave a noticeable tightening upon being worked in this strong gluten wash solution. The fermented gluten did not change. The ordinary gluten and the thoroughly washed gluten showed softening. This suggests that the gluten wash waters work into the meshes of the gluten and cause slippage only where dispersible material is present.

The effect of an oxidizing agent on the physical properties of the same four glutens was studied, as shown in Table VIII.

Substantially no effect upon the fluid properties of these glutens was obtained by manipulating them for 20 minutes in 40 ppm sodium chlorite, except in the case of the gluten produced with ordinary washing. The gluten obtained by thorough washing did not react, thus

TABLE VIII

EFFECT OF OXIDATION ON PHYSICAL PROPERTIES OF GLUTEN

(S = spring; F = flow)

Type of gluten	Unoxidized		Oxidized	
	S	F	S	F
1. Gluten by 5 dispersions	1.38	0.11	1.34	0.12
2. Gluten by thorough washing	1.26	0.16	1.17	0.14
3. Gluten from fermented dough	1.18	0.15	1.15	0.15
4. Gluten by ordinary washing	1.76	0.64	1.11	0.22

suggesting that the material in gluten which reacts to oxidizing agents is soluble in water or removable by yeast.

Summary

The method of testing doughs developed by Halton and Scott Blair (1937) has been modified to simplify the sampling technique, making it possible to work on fermented as well as unfermented doughs and compare the results.

The property of flow, which is the most significant characteristic of doughs, as measured by this method, shows that unyeasted doughs from patent flour containing sugar may soften with time. These doughs upon addition of yeast or of an oxidizing agent lose the property of softening and if treated more heavily they tighten progressively.

Experiments indicate that the reaction of doughs to oxidizing agents is not concerned in any marked degree with the starch, "amylodextrin," or fat portion of the dough, but is located in either the gluten or the water-soluble portion of the dough.

A device was built to test the same characteristics of gluten as were tested on doughs by the apparatus of Halton and Scott Blair. Tests indicate that glutens prepared from unfermented doughs containing no yeast react to oxidizing agents. When prepared from fermenting doughs containing yeast, tight glutens which do not react are obtained. This indicates that the response of gluten to these treatments is similar to that of doughs.

Upon subjecting glutens to thorough washing conditions it was observed that the property of flow could be largely removed from the gluten, indicating that this property is associated with water-soluble constituents.

Glutens subjected to the action of papain undergo a marked progressive softening, opposite to the effects of oxidation. Changes in gluten similar to oxidation are produced by extensive washing.

Water solubles of gluten were further extracted by dispersing mechanically in ¾% salt solution with a Waring Blendor and the gluten collected by centrifuging. When gluten which had been subjected to thorough washing conditions was thus dispersed, a relatively large amount of protein material was rendered soluble. Further, decreasing quantities could be removed on successive dispersions until finally the amount of soluble protein removed by each dispersion reached a constant value, indicating that all of a readily dispersible type of material has been removed and only a true gluten component was now dissolving. This dispersible material showed many of the properties of a proteose. Glutens that had been subjected to washing or to fermentation showed less of this dispersible fraction than ordinary glutens. Glutens receiving fermentation showed less than the washed gluten, indicating that fermentation had removed some of this material, thus possibly explaining some of the tightening effect on dough exhibited by yeast.

Glutens were manipulated in the most concentrated "gluten wash solution" we were able to prepare. The redispersed gluten tightened as a result of handling, just as any gluten ordinarily will when manipulated in salt water. The gluten from yeast fermentation showed no change from the handling. Rewashed or ordinary gluten showed marked softening from this treatment, indicating that the dispersible fraction left in the gluten was softened by the ingredients of the strong gluten wash solution and rendered more fluid so that the meshing of the gluten molecules became less firm and slippage occurred.

The same four glutens were manipulated in salt solution containing sodium chlorite. Substantially no change occurred in any of the glutens except the one subjected to ordinary washing. This gluten exhibited a marked tightening, much greater than would have occurred had not the oxidizing agent been present. This evidence suggests that the material in gluten which reacts to the oxidizing agent is soluble in salt water and can be washed from the gluten by sufficient treatment, and it indicates that the reactive material is found in the more readily dispersible fraction.

Literature Cited

Baker, J. C., and Mize, M. D.
1941 The origin of the gas cell in bread dough. Cereal Chem. **18**: 19–34.
Balls, A. K., and Hale, W. S.
1936 Proteolytic enzymes of flour. Cereal Chem. **13**: 54–60.
Freilich, J.
1941 A method for the determination of reducing matter in flour. Cereal Chem. **18**: 129–137.
Hale, W. S.
1939 The proteinase in wheat flour. Cereal Chem. **16**: 695–702.

Halton, P., and Scott Blair, G. W.
1937 A study of some physical properties of flour doughs in relation to their bread-making qualities. Cereal Chem. **14**: 201–219.
Jørgensen, H.
1936 On the existence of powerful but latent proteolytic enzymes in wheat flour. Cereal Chem. **13**: 346–355.
Kosmin, Natalie P.
1934 Kleberqualität und einige Faktoren, die diese beeinflussen. Mühlenlab. **4**: 110–115.
Landis, Q.
1940 Polariscopic determination of proteolytic activity. Cereal Chem. **17**: 468–472.
Read, J. W., and Haas, L. W.
1937 Studies on the baking quality of flour as affected by certain enzyme actions. IV. Potassium bromate and enzyme activity. Cereal Chem. **14**: 752–765.
Sandstedt, R. M., Jolitz, C. E., and Blish, M. J.
1939 Starch in relation to some baking properties of flour. Cereal Chem. **16**: 780–792.
Sullivan, B., Howe, M., Schmalz, F. D., and Astleford, G. R.
1940 The action of oxidizing and reducing agents on flour. Cereal Chem. **17**: 507–528.

AN IMPROVED METHOD FOR THE VOLUMETRIC DETERMINATION OF SODIUM CHLORIDE IN BREAD

ROY IRVIN

Red Star Yeast & Products Co., Milwaukee, Wisconsin

(Read at the Annual Meeting, May 1941)

Investigation showed that the official method for the determination of sodium chloride in bread gave low and variable results, less than 70% of the salt being recovered when the method as outlined in *Cereal Laboratory Methods* (A. A. C. C., 3rd ed., 1935) was followed. As it was suspected that chloride was being lost through sublimation during ignition, the addition of various alkaline substances to bread crumb prior to ashing was investigated. Reports of studies by others also indicated loss of chloride during ashing of bread crumb and similar substances. Hoffmann, Schweitzer, and Dalby (1940) found that sodium hydroxide prevented the loss of iron through sublimation of ferric chloride during the burning of bread crumb and Kent-Jones (1927) made use of alcoholic sodium hydroxide in the recovery of chloride from flour fat.

In this work the procedure as given in *Official and Tentative Methods of Analysis* (A. O. A. C., 4th ed., 1935) for the determination of chloride in plant materials was taken as a basis for an improvement of the present official method for salt in bread. According to the A. O. A. C. test, sodium carbonate is employed to prevent chloride escape.

Experimental

Preliminary analyses indicated conclusively that the ashing of bread crumb without a fixative agent for chloride would yield low salt values, but that good recovery was possible when the crumb was treated with sodium carbonate or sodium hydroxide before ashing. Less favorable results were obtained with zinc oxide, calcium carbonate, magnesium oxide, and magnesium acetate (alcoholic solution). Several analyses of dried bread crumb, as well as of flour containing added salt, showed an average chloride loss of 30% to 35% when no alkaline substance was added to the samples before ashing, and a 10% to 15% loss when the samples were first treated with calcium or magnesium compounds. In case of the alkaline earths, a single ashing of crumb usually sufficed, but with the sodium compounds it was necessary to leach the charred sample with water or dilute nitric acid before the carbon could be completely burned.

A few determinations of salt in bread were also made by adding three successive portions of 5 ml of 4% alcoholic potash to 3-g samples of crumb during ignition. This was followed by a single filtration of the ash. Although the recovery of chloride by this method was fairly good—being about 97% to 98% of the calculated value—some loss of material occurred through spattering, and only in the case of bread samples made from a no-salt formula was a white ash obtained. When the crumb contained a normal amount of salt, the ash fused badly with the alcoholic potash procedure and showed particles of unburned carbon. The disadvantages of this method might be overcome through further investigation, thus making possible the elimination of the double ashing found necessary with sodium carbonate.

To expedite the analysis of ash of the bread crumb, Caldwell's modification of the Volhard volumetric method for chloride was chosen as a satisfactory procedure. The method as used in this investigation is described by Kolthoff and Sandell (1936). The Caldwell method, which does not require removal of the precipitated silver chloride before back titration with thiocyanate as in the usual Volhard procedure, was found by trial to be sufficiently accurate for the purpose in hand.

Bread for the experimental work was made in the laboratory from two types of formulas, one containing 5% sugar, 3% shortening, and 3% yeast, the other containing 5% sugar, 3% shortening, 5% skim-milk solids, 0.5% malt syrup, 0.3% yeast food, and 3% yeast. The salt content was varied from 0 to 2.25%. The ingredients were carefully weighed for each loaf, all of the dough was used, and the crumb from the dried loaf was quantitatively recovered, thus making it possible to calculate the added salt content of the dried crumb. Chloride blanks for the nonsalt materials were obtained by analysis of

bread made without salt. In Table I, samples 1 and 2 were derived from bread made according to the lean formula and samples 3, 4, 5, and 6 were from bread prepared from the enriched formula. In the case of the no-salt doughs, fermentation time was shortened to prevent excessive loss of materials through rapid yeast action.

TABLE I

SUMMARIZED RESULTS OF SALT DETERMINATION IN BREAD—DRY BASIS

	Sample 1 (blank)	Sample 2	Sample 3 (blank)	Sample 4	Sample 5	Sample 6
	%	%	%	%	%	%
Total percent found	0.116	2.15	0.273	1.78	2.24	2.52
	0.114	2.15	0.283	1.77	2.26	2.52
	0.114	2.14	0.283	—	2.26	—
	—	2.16	—	—	—	—
	—	2.15	—	—	—	—
	—	2.18	—	—	—	—
Average	0.115	2.16	0.280	1.78	2.25	2.52
Net percent, average	—	2.05	—	1.50	1.97	2.24
Calculated percent	—	2.090	—	1.506	1.995	2.241

Prior to analytical work, the glassware employed was calibrated and during the determinations corrections were made for variations in the temperature of standard solutions. Strongly heated sodium chloride of the highest grade obtainable was used as an ultimate standard. Its purity was checked against fused silver nitrate by both the gravimetric and the Caldwell methods.

The modified procedure for the determination of salt in bread is as follows:

Reagents:

1. Silver nitrate. Adjust to 0.05*N* strength by standardizing against 0.05*N* NaCl solution containing 2.923 g pure NaCl per liter.
2. Potassium thiocyanate. Adjust to 0.05*N* by titrating against 0.05*N* $AgNO_3$.
3. Ferric indicator. Saturated solution of ferric ammonium alum.
4. Dilute nitric acid. Dilute the usual pure acid with ¼ volume H_2O. Boil until colorless and then add H_2O to make a 1 + 4 dilution of the acid.
5. Nitrobenzene (cp).
6. Sodium carbonate, 5.0% solution.

Determination: Prepare the samples as directed in Chapter VI, sec. 1, page 83, *Cereal Laboratory Methods*, and determine the moisture in

the air-dried crumb as directed in Chapter VI, sec. 2, page 83. (The air-dried crumb may be first dried as directed in (a) or (b) on page 29, *Cereal Laboratory Methods*, and then weighed.) Weigh 3 g of the air-dried (or completely dried crumb) in a porcelain or platinum crucible and thoroughly wet with 10 ml of 5% Na_2CO_3 solution. Dry at 120°–130°C for about one hour to remove excess moisture, then thoroughly char at a dull-red heat (2–4 hours' ignition). After the crucible is cool, cover the char with water, let stand a few minutes in a warm place, cautiously acidify with 1 + 4 HNO_3 and filter. The filter is well washed with water and small amounts of dilute acid, a total of 15–16 ml of 1 + 4 HNO_3 being used for neutralizing and washing. Place the paper and char back in the crucible, partially dry and then ignite to a white ash at a dull-red heat (30–45 minutes required). Solution of ash, acidification, and filtration are carried out as before except that only 2–3 ml of 1 + 4 HNO_3 is required. The second filtration is added directly to the first. Combined filtrates and washings should not amount to more than about 125 ml. Add 25 ml of 0.05*N* $AgNO_3$, 3 ml of nitrobenzene, and 1 ml of ferric indicator in the order given. Shake thoroughly for about 30 seconds or more and titrate the excess $AgNO_3$ with 0.05*N* KCNS solution to a faint reddish-brown tint that does not fade in 3 or 4 minutes. As the true end point is approached a false end point may occur which disappears upon vigorous shaking.

Calculation:

$$\frac{(\text{ml } AgNO_3 \text{ sol} - \text{ml KCNS sol}) \times 0.002923 \times 100}{\text{weight sample}} = \text{percent salt.}$$

Discussion of Results

The order of accuracy obtainable by the revised procedure is indicated by the results given in Table I. It will be recalled that sample No. 1 constituted a blank for the nonsalt materials in sample No. 2, while No. 3 was a blank for the remaining samples (Nos. 4, 5, and 6).

Portions of the same six samples of bread crumb were submitted to three collaborating laboratories for check determinations by the revised method. The results reported by the collaborators are shown in Table II.

While the comparative results found by the different laboratories do not agree as closely as could be desired, the method as described can be considered a distinct improvement over the official procedure for the determination of salt in bread.

TABLE II

RESULTS REPORTED BY COLLABORATORS: NET PERCENT SALT FOUND—DRY BASIS

Sample	Collaborators			Author	Calculated values
	A	B	C		
	%	%	%	%	%
2	2.04	1.91	2.01	2.05	2.090
4	1.52	1.34	1.46	1.50	1.506
5	1.96	1.88	1.94	1.97	1.995
6	2.27	1.95	2.21	2.24	2.241
			BLANK VALUES		
1	0.150	0.180	0.13	0.115	—
3	0.325	0.260	0.28	0.280	—

Conclusions

The official method for the determination of salt in bread gives low results due to loss of chloride during ashing.

Substantially complete recovery of chloride from bread crumb can be obtained by the addition of sodium carbonate prior to ashing.

Caldwell's modification of the Volhard procedure for chloride is rapid and sufficiently accurate for the analysis of the ash of bread.

It is suggested that the substitution of alcoholic potash for sodium carbonate in this determination be further investigated.

Acknowledgment

The author wishes to express his thanks to collaborators C. N. Frey, E. L. Von Eschen, and F. A. Collatz for the generous assistance rendered by them in this investigation.

Literature Cited

American Association of Cereal Chemists
1935 Cereal laboratory methods (3rd ed.).
Association of Official Agricultural Chemists
1935 Official and tentative methods of analysis (4th ed.). Washington, D. C.
Hoffmann, C., Schweitzer, T. R., and Dalby, G.
1940 Iron content of bread and bread ingredients. Ind. Eng. Chem. (Anal. Ed.) 12: 454–455.
Kent-Jones, D. W.
1927 Modern cereal chemistry (2nd ed.). Northern Publishing Co., Liverpool.
Kolthoff, I. M., and Sandell, E. B.
1936 Textbook of quantitative analysis. Macmillan Co., New York.

REPORT OF THE 1940–41 COMMITTEE ON METHODS OF TESTING CAKE FLOUR

J. W. MONTZHEIMER, *Chairman*

Centennial Flouring Mills Co., Spokane, Washington

(Read at the Annual Meeting, May 1941)

The object of this year's committee on methods of testing cake flour was to determine the value of the A. A. C. C. tentative cake test and the sugar and shortening supplements described in the book of methods for determining the value of cake flour in commercial formulas.

TABLE I

ANALYSIS ON CAKE FLOURS[1]

Sample	Ash	Protein	Moisture	pH	Viscosity
	%	%	%		° *MacM*
W	0.361	7.0	12.5	4.9	32
X	0.36	8.4	12.4	5.2	68
Y	0.405	8.5	12.6	5.3	70

[1] Analysis by V. H. Morris, Federal Soft Wheat Laboratory, Wooster, Ohio.

Three flours from widely separated sections of the country were chosen (Table I). Samples of these flours were submitted to committee members, by whom they were baked according to the A. A. C. C. formula with supplements and scored by the official score card for cake together with the stop grades as proposed by the 1939–40 committee. For average scores on flour submitted, see Table II. Ratings of the flour by committee members are shown in Table III. The same flour samples baked in regular commercial cake formulas and judged by bakers' service men are rated in Table IV.

TABLE II

AVERAGE OF COLLABORATORS' SCORES

Formula and supplement	X			Y			W		
	Av. volume	Av. score	Av. grade	Av. volume	Av. score	Av. grade	Av. volume	Av. score	Av. grade
	cc			cc			cc		
A. A. C. C.	730	81	Good	750	77	Fair	730	82	Good
Sugar 10%	713	79	Good	754	79	Fair	708	80	Good
Sugar 20%	736	79	Good	750	72	Fair	729	74	Fair
Sugar 30%	712	73	Poor	744	68	Poor	715	64	Poor
Shortening 25%	751	83	Good	781	79	Good	761	83	Good
Shortening 50%	742	83	Good	759	77	Good	719	83	Good
Shortening 75%	702	79	Fair	719	77	Fair	662	86	Good
Av. of all tests	726	79.5	—	751	75.5	—	718	80	—

TABLE III

RATINGS ACCORDING TO A. A. C. C. FORMULA

Committee member	1st	2nd	3rd	Comments
Armstrong	W	X	Y	Y increased in volume with added sugar and shortening. Recommended for rich formulas.
Haley	X	W	Y	X for all-round cake purposes. W for cookies.
Mitchell	Y	X	W	Y best volume. X best symmetry.
Montzheimer	W	X	Y	X and W about equal. Y coarse grain and poor color.
Stokes	W	X	Y	Y best for pound cakes. X for medium rich sugar and light loaf. W is best for sponges and angel cakes.
Wade	W	X	Y	Flours all good.

TABLE IV

RATINGS BY COMMERCIAL FORMULAS

	1st	2nd	3rd
White layer	W	Y	X
Yellow layer	W	X	Y
Devil's food	W	X	Y
Pound cake	Y	X	W
Sponge cake	X	W	Y
Angel cake	X	W	Y

The committee feels that the following conclusions may be drawn from this year's work:

1. It would appear that W is best suited for layer and devil's food cake, Y for pound cake, and X for sponge and angel cake.

2. The A. A. C. C. formula with supplements is valuable in predicting the behavior of cake flours in commercial formulas containing shortening.

3. The A. A. C. C. formula does not indicate the suitability of a flour for angel or sponge cakes.

4. Bakers and commercial men rate volume the most important in judging cakes. It has been suggested that our present rating system lays too much importance on cake appearance, color, grain, and crust, and not enough value is placed on volume.

The following suggestions are recommended for the new committee: (1) That this year's project be repeated, except that changes in sugar and shortening should be accompanied by a balance of the other ingredients. This year's committee felt that the present supplements spoiled the cake, because the ingredient ratio was wrong. (2) Further investigation of mixing should be undertaken, including a study of the

right time for adding leavening agents and the use of specific volume as a method of determining the final mixing time. (3) Collaborative studies should be made at various altitudes to check the work done by Mr. Barmore, for correction of the baking powder in A. A. C. C. formulas at various altitudes. (4) This year's committee believes that a great deal of worth-while work could be carried out by local cake committees in the various sections.

Acknowledgment

This year's work was carried out by the following committee members: Donald Wade, Lowell Armstrong, William Haley, W. E. Stokes, R. W. Mitchell, and K. Rourbaugh.

GRANULATION AS A FACTOR IN CAKE FLOUR QUALITY

W. H. HANSON

Commercial Milling Company, Detroit, Michigan

(Read at the Annual Meeting, May 1941)

Researches by capable workers such as Alsberg, Mayer, Ling, Katz and numerous others have added greatly to our knowledge of the wheat starches. The work of the cereal chemists, particularly those who are located within the soft winter wheat area, has accomplished much in evaluating the importance of tests such as hydrogen-ion concentration, viscosity, diastatic activity, and flour granulation when these factors are applied to actual bake shop performance.

A review of the literature on flour granulation indicates that this subject has received a great deal of consideration. Alsberg and Griffing (1925) showed the effect of overgrinding on hard winter and spring wheat varities, and the subsequent effect on loaf volume. Woodruff and Nicoli (1931) summarized the importance of starch gels as affecting the flour quality. Sandstedt, Jolitz, and Blish (1939) indicated the importance of starch in relation to some baking properties of flour, and state that "certain undesirable baking characteristics of some exceedingly hard wheat starches are due to damage to the starch in milling." Jones (1940) summarized the importance of mechanically damaged starch in milling, and its effect upon the diastatic activity of flour. Alexander (1939) suggested a method for determining the granulation of flours in connection with the usual laboratory routine tests which are made.

Hastings (1938) states that "the size of the starch cells of wheat is apparently influenced to a large degree by the variety, growing and ripening conditions, and the protein strength." The size of the starch cell of soft winter wheat is therefore obviously important, in that a

finely milled flour seems better adapted for cake manufacture. It is our opinion that the granulation of a flour may change from one crop to the next, depending upon the growing and ripening season of the grain. If such changes are manifested, it is necessary to make the proper changes in milling to arrive at a satisfactory degree of fineness in granulation comparable to the previous year.

Our tests have indicated that the size of the flour particle is not dependent upon the use of exceedingly fine silk bolting cloths in the flow of a mill. A proper balance of the mill stocks in the bolters and purifiers and selective grinding operations seem more important to the resulting flour. Controlled breaking of the mill stocks is very essential, and should be carefully checked in order to maintain the proper balance. Small sifters are now being used in many mills to good advantage as a means of checking hourly any change which might occur in the grinding operation.

Jones (1940) states that "all flours contain a certain proportion of their starch granules mechanically damaged as a result of the milling process." It seems impossible to mill soft winter wheat flours without dislodging starch granules from the matrix surrounding each particle. The presence of "ghosts" in flour is very important, in that ruptured starch granules should be avoided as much as possible in the grinding operation. While a fine granulation seems to be very desirable, the grinding must not be carried to the extent of mechanical rupture of the starch granule, and a subsequent weakening of the gel strength in cake batters.

Alsberg and Griffing (1925) state that "the strength of the gel is dependent upon the size to which the granules swell, and the volume which they occupy as compared to the total volume in which they are suspended." Tests which have been made show quite conclusively that the gel strength is associated with the size and number of starch granules present in the batter emulsion. Flours which are milled from the same wheats but varying 5% or more in granulation [1] show decided differences in cake volume and internal cake characteristics. It is evident that a coarse flour is less evenly distributed in a batter emulsion, which has a tendency to weaken rather than strengthen the gel structure. When high-ratio formulas are used, it is very important that the flour be finely milled and of the proper analysis and granulation to give the best results.

A survey was conducted primarily to check the granulation of the various flour streams which are selected for definite cake flour types,

[1] There is danger that these statements and quotations may lead to confusion of "starch granuel size" and "granulation." These terms are in no way synonymous. Starch granule size refers to the size of the individual starch granule which is not determined by grinding or bolting. Granulation refers to the size of the flour particles and is determined by milling and bolting procedures. A flour granule contains a large number of starch granules.

and to note any improvement by using finer silks as affecting the flour quality. The Ro-Tap testing sieve shaker was used in the experiment, and the percent of stocks remaining on the sieves after each 10, 20, 30, and 45 minute operation was recorded. Carmichael cloth cleaners were used on each sieve in order to facilitate the bolting operation. The moisture present in the flour streams tested seemed to be a critical factor, and in order to eliminate this as much as possible all the samples were dried to approximately 11%. It was found necessary to continue the bolting operation on many of the lower grade streams for 60 minutes, and then some difficulty was experienced in obtaining good replications. This was due in part to the soft gummy and flattened condition of the clear and low grade stocks. In place of the standard silk bolting cloth which is normally used in the flow of a mill, we substituted metal sieves which were permanently fastened to eight-inch metal frames. The average dimension of the aperture opening and mesh of the metal sieves are given in Table I together with the equivalent in silk bolting cloth.

TABLE I

SIEVE DIMENSIONS

Mesh and opening—wire cloth	Equivalent openings to silk
165 mesh Dur-Loy bolting cloth, 0.0042″ opening	12XX or 13XX standard silk
230 mesh Dur-Loy bolting cloth, 0.0029″ opening	21XX standard silk
250 mesh Phosphor Bronze cloth, 0.0024″ opening	25XX standard silk

Courtesy W. S. Tyler Company.

The results shown in Table II indicate the variation found in a few of the mill streams listed. We have excluded from this series many of the higher-ash streams which are normally classified as clear and low

TABLE II

GRANULATION OF FLOUR STREAMS OBTAINED ON RO-TAP SIEVE SHAKER IN 45-MINUTE OPERATION

Mill stream	Ash (%)	Granulation (%) (throughs of 250 mesh)
1st middlings	0.275	97.0
2nd middlings	0.280	96.0
3rd middlings	0.295	93.0
4th middlings	0.325	89.0
5th middlings	0.350	84.0
6th middlings	0.385	80.0
7th middlings	0.505	72.0
Sizings	0.305	97.0
1st break flour	0.400	98.0
2nd break flour	0.360	97.0
3rd break flour	0.355	97.0
4th break flour	0.445	83.0
5th break flour	0.620	70.0

grades. The mill streams tested seemed important in that the lower-ash streams which comprised approximately 40% to 50% of the total flour gave a very satisfactory granulation. The ash test is given only as an indication of the refinement of the mill streams, as this factor cannot be correlated with the granulation. This is clearly shown when we compare the break and middling flours of nearly the same ash content. The break flours, which are usually low in viscosity, do reflect a satisfactory granulation, although they are usually somewhat higher in ash depending upon the flow of the mill. The low-ash middling flours are higher in viscosity, and should receive due consideration if any changes are contemplated in the flow of the mill in order to obtain a finer flour.

The results of Table II are interesting in comparing flours of different extraction, in that various streams which may be included will reflect a definite trend in granulation. For this reason we feel that a short patent cake flour of 40% to 50% extraction is finer than flours which have been milled to a 95% extraction. This may partially explain the preference of bakers who demand short-patent cake flours over the longer extractions. It is not uncommon during the year to obtain some wheat which is hard and flinty in character. Wheats of this type when milled will reflect a notable change in the granulation of the flour. It is for this reason that great care should be exercised in the purchase of wheat so that it will give the desired characteristics when milled into flour. Abnormal ripening conditions of the wheat during the growing season, variety, and a number of other factors may change materially the granulation from one crop to the next. It is important therefore to check the flours very carefully, and to maintain a definite standard on all grades.

TABLE III

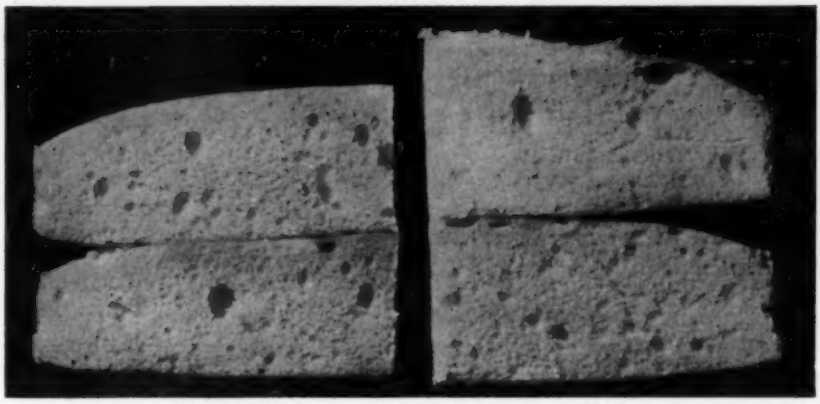
ANALYSIS OF FLOUR STOCKS BOLTED FROM A SHORT-PATENT CAKE FLOUR IN 45-MINUTE OPERATION OF RO-TAP SIFTER

Analysis	Short-patent flour (standard)	Stock remaining on 250 mesh	Stock through 250 mesh
Ash, %[1]	0.345	0.355	0.342
Protein, %[1]	7.90	9.50	7.50
Viscosity, No. 1.[2]	23	76	20
Viscosity, No. 2.[2]	31	85	25
pH	5.00	5.10	5.00

[1] Ash and protein reported on 15% moisture basis.
[2] Viscosity reported by No. 1 (no-time method) and No. 2 (1-hour digestion method) in degrees MacMichael.

The data shown in Table III indicate a difference between the analysis of the stock remaining on a 250-mesh sieve and the very fine flour which was bolted through the same mesh. This difference in

granulation may be due to the fact that in a wheat mixture a very small percentage of hard vitreous kernels present are not reduced properly in the milling process. It is for this reason that careful selection of the wheat should be made before undesirable grades have been purchased.



3 4

Fig. 1. Shows the results obtained by the cake test of the stock remaining on a 250-mesh (cake No. 3) as compared with the fine stock bolted through a 250-mesh (cake No. 4).

Cake tests shown in Figure 1 indicate the relative importance of granulation in cake flours. The recommended tentative basic-cake formula of A. A. C. C. was used.

Summary and Conclusions

The granulation test has been found useful in standardizing the degree of fineness to which flours are milled, and is therefore recommended as a laboratory routine test. Granulation may be considered as one of the factors which seem to influence the baking characteristics of a cake flour, although not essentially the most important one.

Short-patent cake flours seem better suited for high-ratio formulas, primarily because of the better quality of the streams selected and the finer granulation which is present in these streams.

The character of the flour gluten and percent of flour extraction, protein, viscosity, and pH are all obviously important, but these alone will not insure optimum baking results unless the flour has been milled to a predetermined degree of fineness.

The granulation of a flour seems more dependent upon the proper balance of the flour streams in the bolters and purifiers, than upon the fine bolting silks which are used in the flow of the mill.

The comparative effects of differences in granulation of the mill streams selected are in accordance with the observations of those who have previously contributed to this subject.

Literature Cited

Alexander, G. L.
1939 Soft-wheat testing problems. Cereal Chem. **16**: 197–208.
Alsberg, C. L., and Griffing, E. P.
1925 Effect of fine grinding upon flour. Cereal Chem. **2**: 325–344.
Hastings, P. O.
1938 Starch as a factor in cake flour quality. Association of Operative Millers, Bulletin 31, p. 922.
Jones, C. R.
1940 The production of mechanically damaged starch in milling as a governing factor in the diastatic activity of flour. Cereal Chem. **17**: 133–169.
Sandstedt, R. M., Jolitz, C. E., and Blish, M. J.
1939 Starch in relation to some baking properties of flour. Cereal Chem. **16**: 780–792.
Woodruff, S., and Nicoli, L.
1931 Starch gels. Cereal Chem. **8**: 243–251.

REPORT OF THE 1940–41 COMMITTEE ON TESTING BISCUIT AND CRACKER FLOURS

H. J. LOVING, *Chairman*

The Kroger Food Foundation, Cincinnati, Ohio

(Read at the Annual Meeting, May 1941)

The 1939–40 Committee on Testing Biscuit and Cracker Flours recommended that (1) further work on cracker flours be suspended until the statistical study could be prepared covering the five years' work done by the committee on that subject, and (2) that work be done on biscuit or cooky type flours in order to bring such tests up to the present status of tests on cracker flours (Simmons, 1941). The statistical study referred to has been prepared and submitted to the committee by one of its members, Mr. Tarnutzer. After appropriate consideration by the committee, it will be placed in final form and submitted for publication.

For the evaluation of cooky flours, the work of this year's committee has taken the form of a collaborative study of the formula and procedure proposed by Alexander (1933).

The flours chosen on which to conduct the cooky-test study were three soft winter wheat flours from the Middle West, the intermediate being a blend of equal parts of the stronger and weaker. Analyses of the three flours employed appear in Table I.

It has been suggested that the performance of cooky flours may be related to particle size. Table II presents data obtained from a granulation study of the three flours. The results seem significant, but in view of strength differences as revealed by protein and viscosity determinations, it is impossible to properly evaluate the influences of particle size distribution.

TABLE I

ANALYSIS OF FLOURS

	Flour No. 1	Flour No. 2	Flour No. 3
Ash (15% moisture basis) %	0.398	0.393	0.402
Protein (15% moisture basis) %	7.81	8.54	9.07
Viscosity, no-time method[1]	27	52	72
Viscosity, 60-min digestion[1]	39	65	91
Viscosity, 2-g protein	49	71	87

[1] Sample weights adjusted to 20 g at 15% moisture.

TABLE II

GRANULATION OF COOKY FLOURS OBTAINED IN 50 MINUTES WITH RO-TAP SIEVE SHAKER

	Granulation test—%		
	Sample No. 1	Sample No. 2	Sample No. 3
Moisture, %	10.50	10.40	9.80
On 165 mesh, 10 min	X	X	X
On 165 mesh, 20 min	X	X	X
On 230 mesh, 10 min	8	10	13
On 230 mesh, 20 min	2	5	7
On 250 mesh, 10 min	70	71	68
On 250 mesh, 20 min	48	53	48
Through 250 mesh, 30 min	73	66	65
Through 250 mesh, 40 min	86	80	79
Through 250 mesh, 50 min	93	88	84

The flour which was identified to the collaborators as No. 1 was a 94% flour from Michigan white wheat running approximately 9.0% protein. The flour identified to the collaborators as No. 3 was a 95% extraction flour from high-protein Ohio soft red winter wheat. Flour No. 2 was a thorough blend of equal parts of flours No. 1 and No. 3. All three flours were unbleached.

Samples of these three flours, and along with them instructions for applying the cooky formula previously referred to, with slight modifications, were submitted to the committee members. In order to cover sufficient ground to make the study worth while, five possible variables in formula or procedure were chosen, and each of these variables was assigned to either one or two committee members for investigation and possible change in the cooky test.

Dunn (1933) expressed some disappointment in laboratory tests of flours in cookies where "spread" was evaluated. He stated that "In spite of all we could do, there was a lack of uniformity in the spread of these cookies, as well as in the contour and general appearance, which was so great that fine distinctions between flours could not be drawn." It was the idea of the committee that it should be possible

to make adjustments in the formula employed to permit more sensitivity, or sharper differentiation between flours of different strengths and thus minimize such lack of uniformity as familiarity with the test would not prevent. Each collaborator investigated a variable and at the same time conducted bakes on the test as submitted, which gave a common basis for comparison among all operators.

Instructions for conducting the laboratory cooky tests were as follows:

Formula:

Baker's Special granulated sugar	130 g
Hydrogenated fat	64 g (75° F)
Whole egg (fresh)	26 g
Ammonium carbonate	0.5
Soda	2.5
Skim milk suspension	40 ml
Flour	224 g
Salt	2.1 g

28.2 grams of spray-powdered skim milk are mixed into 150 ml of distilled water to make the skim milk suspension.

Procedure: Cream sugar, shortener and soda three minutes, cutting down after each minute (Kitchen Aid Model G, second speed, 128 rpm, or equivalent).

Add eggs slowly during one minute in low speed (low speed on Kitchen Aid, 62 rpm). Scrape down. Mix in second speed for one minute.

Dissolve salt and ammonium carbonate in the skim milk suspension. Add skim milk suspension during one minute in low speed. Scrape down. Mix one minute in second speed. Add whole quantity of flour, mix for two minutes in low speed, cutting down after each half minute.

Place small handfuls of batter at six well spaced points on a cooky sheet so that the cookies when cut will be about two inches apart. Make sure that each handful of batter is coherent and not composed of different scraps pressed together, as this latter practice tends to produce imperfect cookies. Lay wooden strips 7 mm in thickness along each side of the cooky sheet and roll the batter out with rolling pin to this height. Cut a cooky in the center of each piece with the cutter provided (lower part of ointment tin) which has a diameter about 60 mm. Remove scrap and discard, leaving cookies in place ready to be baked.

Bake cookies at 400°F for 10 minutes. On removal from the oven immediately lift cookies from pan to cooling rack or absorbent paper.

After 30 minutes inspect the cookies and measure thickness and width. Thickness can be best measured by piling six atop one another and averaging the height. Average diameter should be obtained by making two measurements of diameter on each cooky.

The spread factor W/T should be computed, W being average diameter and T the average thickness. The greater this factor, the more spread possessed by the cookies.

A score form was provided each collaborator, requiring qualitative evaluation of symmetry, regularity of edge, top color, top grain, bottom appearance, and grain and texture.

Discussion of Results

C. C. Armuth investigated sugar levels over a range of minus 10% of the amount given in the formula to plus 10%. Distinctly more spread was seen at the 10% higher sugar level, and distinctly less spread at the lower sugar level, as compared with the standard formula.

The mixing stage after addition of flour was given in the instructions as two minutes in low speed on a Model G Kitchen Aid (about 62 rpm). The variable investigated by C. E. Bode constituted mixing times of one to three minutes, or a variation of plus or minus 50%. Widest differentiation between the three flours was exhibited at 1½ minutes of mixing time. Preference was expressed by this collaborator for the cooky made at 2½ minutes of mixing time. He also favored the use of second speed in this operation as yielding cookies easily differentiated as well as being expedient in preparation. The actual amount of spread obtained did not vary greatly with the degree of mixing employed.

Variation in liquid content of the formula from minus 10% to plus 10% of the quantity called for was investigated by Miss Brown. Correct differentiation between the flours was made in practically every series of bakes, although the influence of changing liquid level was not as pronounced in spread as was expected by this collaborator.

W. H. Hanson varied mixing time at levels of plus 25% and plus 50% of the recommended treatment. This collaborator stated that he could easily identify the weakest flour, but had some difficulty in his differentiation by cooky tests between flours No. 2 and No. 3, and also that the cooky spread seems very definitely associated with wheat variety to a large extent. Work with the granulation test may reveal that it is a factor in milling the same variety of wheats, as reflected by the cooky test. As was noted in the case of the other collaborator investigating the same variable, the changes in mixing time did not greatly affect the absolute spread experienced.

Liquid variations as well as oven conditions were investigated by T. E. Hollingshead. The liquid levels utilized were from minus 10% to plus 20% of the quantities originally specified. Other conditions being equal, this collaborator found that the higher the liquid level, the more the spread, and conversely. As was noted by at least one other operator, Hollingshead noted that much of the spring taken on by the cookies in the oven was lost when they were removed and allowed to cool. This frequently left a lumpy or irregular top which contributed to variations in measuring average thickness. Bakes were made wherein top heat was used for the last five to seven minutes of oven treatment, which successfully baked the cooky tops and caused them to retain the complete spring gained in the oven, even after cooling. It was stated by this collaborator that the addition of 15% additional water to the original formula gave the maximum spread consistent with good texture.

The investigation of the influence on spread and pH contributed by variations in soda was conducted by H. M. Simmons. As would

be expected, increase of soda increased alkalinity, and decreased soda had the opposite effect. Changes in soda level made some modifications of spread (width and thickness) and crispness. Characteristics such as top color, bottom appearance, or shape seemed to be unmodified. In general, spread was greater with increases of soda and less with decreases of soda. It was the recommendation of this collaborator that the soda in the formula be maintained at the stipulated level.

Shortener quantities were varied from plus 20% to minus 30% of the standard formula by C. A. Tarnutzer. In addition to the regular score factors suggested by the committee chairman, this collaborator also included some others. It was concluded that the count per pound would not correlate with differences in flour strength. Tarnutzer was quickly able to point out flour No. 1 as a medium to weak cooky flour. Proper designation of the strength ranking of flours No. 2 and No. 3 was also made, but some confusion was apparent in the light of the cooky test results. It was the conclusion of this collaborator that as a single test for flour strength, the cooky test with which the committee was working should be modified to the leanest level here investigated, namely a reduction in shortener content of 30%.

Sugar variation from plus 10% to minus 15% of the recommended formula was investigated by H. O. Triebold. It was his conclusion that the standard formula minus 10% or minus 5% of sugar was the most useful in differentiating the three flours.

Summary and Conclusions

Of the ten series completed with the recommended formula and procedure, six ranked the three flours in order of their strength according to protein and viscosity determinations (Table III). Twenty-five

TABLE III

SUMMARY OF SPREAD FACTORS ON TEN MIXES COMPLETED WITHOUT VARIATION OF STANDARD CONDITIONS

Collaborator	Flour No. 1			Flour No. 2			Flour No. 3		
	Av W (*mm*)	*Av T* (*mm*)	*Av W/T*	*Av W* (*mm*)	*Av T* (*mm*)	*Av W/T*	*Av W* (*mm*)	*Av T* (*mm*)	*Av W/T*
Armuth	82.0	11.17	7.34	84.0	11.33	7.41	84.0	10.5	8.00
Armuth[1]	84.0	10.33	8.13	82.0	12.83	6.41	81.8	12.0	6.81
Bode	84.4	11.8	7.15	83.3	12.33	6.75	81.0	13.17	6.15
Brown	77.3	12.0	6.4	77.2	12.75	6.1	72.2	14.2	5.3
Hanson	88.7	12.0	7.39	85.4	12.67	6.74	83.4	13.33	6.26
Hollingshead	83.1	9.3	8.94	79.4	9.7	8.19	80.1	10.5	7.73
Simmons	78.0	10.2	7.67	77.5	11.7	6.65	77.0	11.3	6.87
Simmons	80.3	11.7	6.88	79.6	13.0	6.11	78.8	13.0	6.06
Tarnutzer	81.9	11.94	6.86	80.3	13.61	5.90	80.0	14.20	5.63
Triebold	83.1	12.1	6.9	98.1	11.2	8.8	82.5	11.3	7.3

[1] Different oven employed.

series of the total of forty-four (56.9%) properly ranked the three flours. Most of the exceptions where the proper order was not apparent from the cooky test arose in a confusion of flours No. 2 and No. 3. This will be shown by the fact that 11 of the 44 bakes completed would rank the flours in ascending order of strength as follows: No. 1, No. 3, No. 2.

While admittedly this differentiation between flours varying distinctly in strength is far from being as accurate as certain of our other tests, it is felt that some promise is shown, particularly when we take into account the fact that most of the collaborators were entirely unfamiliar with the test at the beginning of the year's work. Almost without exception each collaborator expressed belief in the value of the test as one suitable for the evaluation of the cooky-making properties of soft wheat flours.

Taking the comments of the various collaborators regarding the variable investigated in each case, we can conclude that modification of the original formula to give greater sensitivity and sharper differentiation between flours of varying strengths could be expected by employing a higher moisture level and by reducing the quantity of shortener and perhaps sugar. It seems to be indicated that little or nothing would be gained by modifying the soda percentage and pH, and indications are that mixing time is about right if these same mixer speeds are employed. However, some interest was expressed in reducing the amount of time for completing the mix by substituting lower times with higher speeds. Some success along this line was reported by Bode and Simmons.

In more than one case objection was expressed to the fact that cookies from the oven immediately developed uneven top shrinkage, leaving a lumpy or irregular "profile," as it was expressed in the cooky inspection report submitted by one of the collaborators. The use of top heat in the oven for the final minutes of baking properly surmounted the criticism and left the entire oven spring in the cooled cookies to be evaluated in the spread factor, which was obtained by dividing the average diameter by the average thickness of six cookies. One collaborator also recommended that the exact quanties of milk and water always be weighed for each batch, inasmuch as proper volume measurement was interfered with by the foaming properties of the suspension.

Recommendations

The work of the committee for this year provided too small a body of data to permit entirely valid conclusions as to the value, reliability, and sensitivity of the tests investigated.

The committee recommends at least one more year of purely laboratory work along these same lines, modified in the direction of higher liquid and leaner formulas generally. It is obvious that greater familiarity will be necessary with this test before it will be a reliable instrument for evaluating flour quality for functions where cookies of the type exhibiting spread are desired.

Acknowledgment

The chairman of this committee expresses here his appreciation of the excellent cooperation afforded by the eight active members of the committee. Expression of appreciation is also made of the work done by E. G. Kornreich of the Kroger Food Foundation and William T. Hanks of the Technical Institute.

Literature Cited

Alexander, G. L.
1933 The results of bleaching Michigan soft winter wheat cake flours by the Brabender electric bleaching apparatus. Cereal Chem. **10**: 623–626.
Dunn, J. A.
1933 Testing biscuit and cracker flour. Cereal Chem. **10**: 628–631.
Simmons, H. M.
1941 Report of the 1939–40 committee on testing biscuit and cracker flour. Cereal Chem. **18**: 252–257.

REPORT OF THE 1940–41 COMMITTEE ON TESTING SELF-RISING AND PHOSPHATED FLOURS

ELMER MODEER, *Chairman*

The St. Joseph Testing Laboratories, Inc., St. Joseph, Missouri

(Read at the Annual Meeting, May 1941)

As the major problem for the 1940–41 year, the committee decided to investigate the practicability of adapting the present biscuit-baking test, by modifying either the formula or the procedure, to enable anhydrous calcium acid phosphate, as well as the hydrated form, to be used. Secondarily, the committee sought further data regarding the present baking test and scoring plan as a means of intra-laboratory characterization of self-rising and phosphated flours.

In the past several years, the use of anhydrous calcium acid phosphate as a leavening agent has become commonplace, so that if possible the ability to interchange the two phosphate forms would be a great convenience.

Accordingly, a series of self-rising flours was prepared, one containing regular hydrated phosphate, in the usual official formula, the others containing varied amounts of anhydrous phosphate, with soda, of course, in proportion. A single-base flour was used throughout, a pure soft Missouri standard patent being selected as a representative type. The analysis of the flour was 9.2% protein, 0.39% ash, viscosity

88° McM, and pH 5.68. It was intended that the flour be bleached with chlorine and benzoyl peroxide, but it was not discovered until after the project was started that the chlorine had been omitted, which doubtless accounts for the relatively low score by the regular method.

The regular hydrated formula sample, designated No. 1, was prepared according to the formula of Walter (1936), namely 1.87% calcium acid phosphate hydrate, 1.5% sodium bicarbonate, 2% sodium chloride based on flour weight.

Samples designated No. 2, No. 3, and No. 4 contained, respectively, 1.5%, 1.3%, and 1.1% anhydrous calcium acid phosphate, and 1.25%, 1.08%, and 0.91% soda, the first being the commercially recommended percentage. All three contained 2.25% salt.

The composite results of the six reporting collaborators are shown in Table I.

TABLE I

COMPOSITE BISCUIT SCORE

	Standard	Samples			
		1	2	3	4
Grain	10	8.0	8.8	8.7	8.3
Tenderness	10	8.0	9.7	9.5	8.5
Flavor	20	18.0	18.1	18.5	18.0
Crumb color	20	16.3	17.9	17.7	17.1
Volume	40	42.6	48.0	45.9	44.2
Total score	100	92.9	102.5	99.3	96.1
pH	—	7.25	7.31	7.23	7.14
Oven loss, %	—	13.2	13.3	12.9	12.2
Specific volume	—	2.13	2.40	2.33	2.21

The committee is in general agreement that the No. 2, No. 3, and No. 4 bakes are not in agreement with No. 1 in any important respect, and actual differences in characteristics are greater than can be revealed in tabulated columns of figures. The handling characteristics of the doughs were different; in general, anhydrous formula doughs are more critical in time-relationships during mixing, rolling, and cutting, contributing to a greater experimental variation. This is evidenced by a range in specific volume among the collaborators of 0.45 for the regular formula, and 0.48, 0.62, and 0.71 for No. 2, No. 3, and No. 4.

In view of the widely different behavior of the two phosphates, it was not deemed necessary to investigate formally a modification of procedure as well as of formula. Because the specific gravity of anhydrous phosphate doughs is greater than hydrated doughs, the possibility of modifying dough thickness by altering roller stick height to bring results into closer agreement was at first considered. Gookins and

Barackman [1] have both studied the problem. The dissimilar leavening action of the two phosphates, as in the case of formula modification, makes it impossible for equal results to be achieved by a simple modification of this nature.

Variation in scores and analytical data, in general, was normal. With the exception of one collaborator, pH determinations were close, and total biscuit scores agreed to an extent comparable to past years. The factor which showed the widest spread, and which in turn doubtless affected biscuit characteristics, was oven loss. The collaborative data for oven loss are shown in Table II.

TABLE II

OVEN LOSS

Collaborator	Samples				Average
	1	2	3	4	
	%	%	%	%	%
A	9.4	8.7	7.8	7.9	8.4
B	15.1	15.0	14.9	15.7	15.2
C	7.4	9.3	7.1	8.5	8.1
D	11.9	11.1	10.3	9.1	10.6
E	16.4	16.6	15.4	16.7	16.3
F	19.0	19.3	17.6	19.5	18.9
Average	13.2	13.3	12.9	12.2	—

This wide variation in oven loss is symptomatic of oven differences which unquestionably affect biscuit character in all respects. It was shown by the 1939–40 committee (Gookins, 1941) that baking to a uniform oven loss did not improve biscuit volume agreement among laboratories. It appears logical, however, that while volume and loss are not directly related, they are related to the same thing, oven environment. Baking to a constant oven loss would not correct all oven differences any more than, for example, baking yeast bread to a common crust color would compensate for all differences in bread ovens.

Conclusions

From a study of hydrated and anhydrous calcium acid phosphate in self-rising test flours the committee finds that the results indicate the characteristics of the two phosphates are much too different for interchangeable use, by modifying either formula or method, so that it is recommended that for intra-laboratory checking for intrinsic self-rising properties of flour, hydrated phosphate and the present recommended method be employed. Secondarily, the results of the committee's collaborative baking and scoring indicate that oven differences,

[1] Private communications.

especially as shown by oven-loss percentages, remain as one of the chief causes of variation among laboratories, a fact which may profitably engage the attention of future committees.

Acknowledgments

Members of the current committee were Lowell Armstrong, R. A. Barackman, O. E. Gookins, Jr., Art King, Elizabeth McKim, F. R. Schwain, and Elmer Modeer. Special acknowledgment is due Mr. Gookins of the committee and Kenneth Spiers of the St. Joseph Testing Laboratories for able advice and assistance.

Literature Cited

Gookins, O. E.
1941 Report of the 1939–40 committee on methods of testing self-rising and phosphated flours. Cereal Chem. **18**: 258–261.
Walter, H. G.
1936 Report of the 1934–35 committee on methods of testing self-rising flours. Cereal Chem. **13**: 722–723.

THE APPLICATION OF VARIOUS BAKING TEST METHODS TO THE EVALUATION OF SOFT WHEAT

J. A. Shellenberger, Paul W. Hodler, and C. A. Nelson

The Mennel Milling Company, Toledo, Ohio

(Read at the Annual Meeting, May 1941)

Baking tests for the appraisal of soft wheat flours are numerous. *Cereal Laboratory Methods* (1935) of the A. A. C. C. outlines baking procedures for bread, cakes, biscuits, and pie crust. The cooky test (Alexander, 1933) should also be included in this list. Theoretically, flour quality should be evaluated by testing the flour for each specific baking purpose. This idealistic concept has many practical limitations. For example, many cereal laboratories are required to estimate the quality of a large number of wheats in a limited period of time, and this precludes an exhaustive study of each flour sample. It is known that each of the baking tests has some merit for evaluating flour, but it is questionable which one, if any, of the tests is capable of providing an adequate index of soft wheat quality. There is need for information which will relate and compare the various baking procedures. The ideal test would differentiate adequately between samples and also would indicate the suitability of the flour for specific commercial use.

The present investigation was undertaken for the purpose of making a comparison between the various baking tests on a series of flours milled from soft wheats. The wheat samples studied were known to differ genetically and to have been grown in different regions.

One of the major considerations in baking tests on experimentally milled wheat samples is to bring the flour to such a condition that its full baking potentialities will be evident. Failure to do this may lead to erroneous conclusions. In this study we have attempted in each case to treat the experimentally milled flour in a manner which would make it comparable with commercial flours used for similar baking purposes. A constant effort was made to test each flour under favorable conditions regarding age, moisture, bleach, and pH for each particular baking procedure.

Description of Material

Twelve samples of soft wheat were obtained from Indiana, Michigan, and Ohio. Ten of these samples were of known variety and two were mixtures from commercial elevators. Two of the samples were soft white wheat and the other ten were soft red winter. The samples are not designated according to variety, since too few tests were conducted to warrant opinion concerning the several varieties included. Thorough baking tests of soft wheat varieties have already been reported by Fifield, Bode, and Bayles (1936) and Bayfield and Shiple (1937).

The wheat samples all had fairly high test weights, the usual protein range, and did not vary greatly in ash content. The samples were normal in all respects. This information is recorded in Table I.

TABLE I

DESCRIPTION OF THE WHEAT AND FLOUR SAMPLES[1]

Reference no.	Wheat			Flour				
	Class	Test weight	Protein (N ×5.7)	Protein (N ×5.7)	Ash	Viscosity	Diastatic activity	Gassing power
		lbs	%	%	%	°*MacM*	*mg*	*mm*
A	SRW	61.2	9.3	8.02	0.35	76	81	166
B	SRW	62.2	9.7	8.13	0.38	91	171	255
C	SRW	62.0	9.9	8.23	0.37	62	122	214
D	SRW	60.0	9.1	7.68	0.32	61	91	185
E	White	60.1	9.0	7.29	0.34	35	97	185
F	SRW	61.0	10.3	8.23	0.32	55	106	214
G	SRW	61.8	10.3	8.34	0.33	96	109	194
H	SRW	61.6	9.5	7.74	0.38	70	90	186
I	SRW	60.5	11.2	9.03	0.34	80	94	167
J	White	60.0	10.5	8.26	0.34	32	88	183
K	SRW	60.4	9.8	7.93	0.36	69	98	195
L	SRW	59.8	9.4	7.73	0.38	58	88	148

[1] Results reported on 15% moisture basis.

The samples were milled on a Buhler mill (Ziegler, 1938) and a fairly short patent flour removed. The flow of the mill was modified

from the original by the addition of scalping screens on the first and second reduction systems. These scalpers played an important part in producing low-ash flour without the necessity of taking too short a patent for practical purposes. Flour yields of approximately 60% were obtained. In Table I are recorded the protein, ash, viscosity, diastatic activity, and gassing power of these flours.

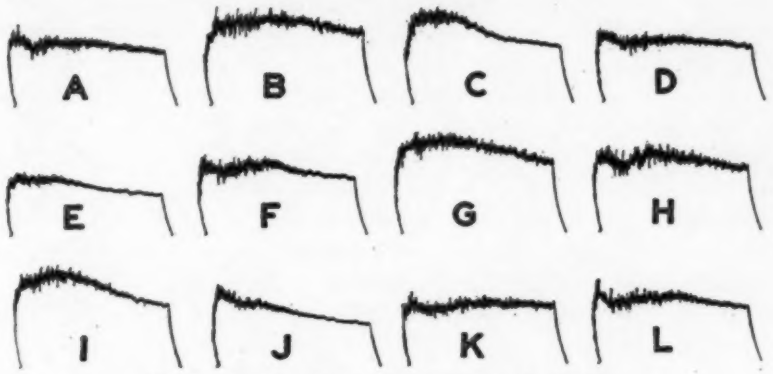


Fig. 1. Micro recording dough mixer curves of soft wheat.

In Figure 1 are shown the Swanson-Working micro recording dough-mixer curves for the various samples. Soft-wheat curve characteristics have not been stressed to the same extent as have the characteristics of hard-wheat curves. Limitations of the mixing and damping mechanisms on the original recording dough mixers made these machines much less flexible for soft-wheat studies than the present type of micro recording dough mixer. It will be noted that the curves approach in magnitude and shape the curves produced from hard wheat flour by Shellenberger (1940). This is the result of using a weaker damping force in order to magnify differences in the curves which would not otherwise be evident. It is readily observed that the physical dough properties of this series of samples vary over a wide range, and it appears that the recording dough mixer might be extremely useful for differentiating between soft wheat varieties. For example, samples *A* and *B* have nearly the same protein content and are both soft red winter wheats, yet their mixing-curve characteristics are decidedly different. Bailey (1940) has recently discussed the value and limitations of physical dough testing methods.

Experimental

Bread-baking tests: The portions of the flour used for the bread-baking test were bleached with a maturing agent and benzoyl peroxide. The diastatic activity of each sample was adjusted by the addition of

malted wheat flour to approximately 135 mg of maltose as determined by the Blish and Sandstedt (1933) method. As indicated in Table I, the flours originally varied from 81 to 171 mg maltose and the corresponding gassing power varied from 148 to 255 mm pressure. Sherwood and Bailey (1926) called attention to the importance of diastatic activity control and its relation to the baking test. More recently, a number of investigations (Landis and Frey, 1933, and Landis, Frey, and McHugh, 1935) have stressed the desirability of eliminating diastatic activity as a variable factor in test baking.

In accordance with the foregoing suggestions, all the flours used in these baking tests, with the exception of sample *B*, were adjusted to approximately the same diastatic activity. Sample *B* had an initial value of 171 mg of maltose, which is unusually high for soft-wheat flours. It was not deemed advisable to adjust all the samples to this level; therefore, this one flour is out of harmony with the others in respect to diastatic activity and gassing power.

The bread-baking procedure followed in most respects the suggestions made by Bayfield and Shiple (1937) for adaptation of the A. A. C. C. basic bread-baking test for soft wheat evaluation. Four percent sugar, 2% shortening, and 0.50 mg of bromate were used, and the doughs were mixed for two minutes in the Swanson-Working mixer. It has been shown by Shellenberger (1938) that more than a one-minute mixing period is required to incorporate completely the dough ingredients. A National Manufacturing Company sheeting roll was used to punch the doughs and also to sheet the doughs before panning.

TABLE II

COMPARISON OF BAKING RESULTS

		Bread		Cake		Biscuit		Cooky			Pie
Reference no.	Absorption[1]	Loaf volume	Bread score	Cake volume	Cake score	Biscuit sp vol	Biscuit score	Average diameter, *W*	Average thickness, *T*	Ratio of *W/T*	Pie crust score
		cc		*cc*		*cc*		*mm*	*mm*		
A	51.7	565	91	705	96.0	36.1	78.1	78.6	14.0	5.62	68.0
B	52.5	525	87	695	95.0	33.8	74.8	80.0	14.0	5.71	74.5
C	51.4	505	87	732	95.9	36.6	79.1	74.6	15.0	4.97	68.0
D	51.3	535	89	675	97.6	35.9	75.9	79.4	13.4	5.93	74.0
E	50.0	500	83	635	96.5	37.7	80.2	82.0	12.8	6.41	77.5
F	50.1	565	91	738	96.9	36.3	77.3	81.4	13.8	5.89	75.0
G	52.3	530	88	717	96.8	35.2	77.0	78.8	14.8	5.33	74.0
H	51.1	545	88	730	96.0	35.9	76.7	78.6	15.0	5.24	77.0
I	51.2	595	90	702	95.8	35.7	77.5	79.2	15.2	5.21	71.5
J	49.4	445	78	685	96.3	35.0	77.1	81.6	13.8	5.92	78.5
K	51.5	540	88	745	96.8	34.3	75.3	81.0	15.0	5.40	70.5
L	51.4	540	88	680	96.5	35.9	76.7	79.2	14.0	5.66	75.0

[1] On 15% moisture basis.

Preliminary bakes were used to determine the optimum absorption for each flour sample.

In Table II are recorded the baking results obtained from the twelve flours, and pictures of the bread are shown in Figure 2. The bread

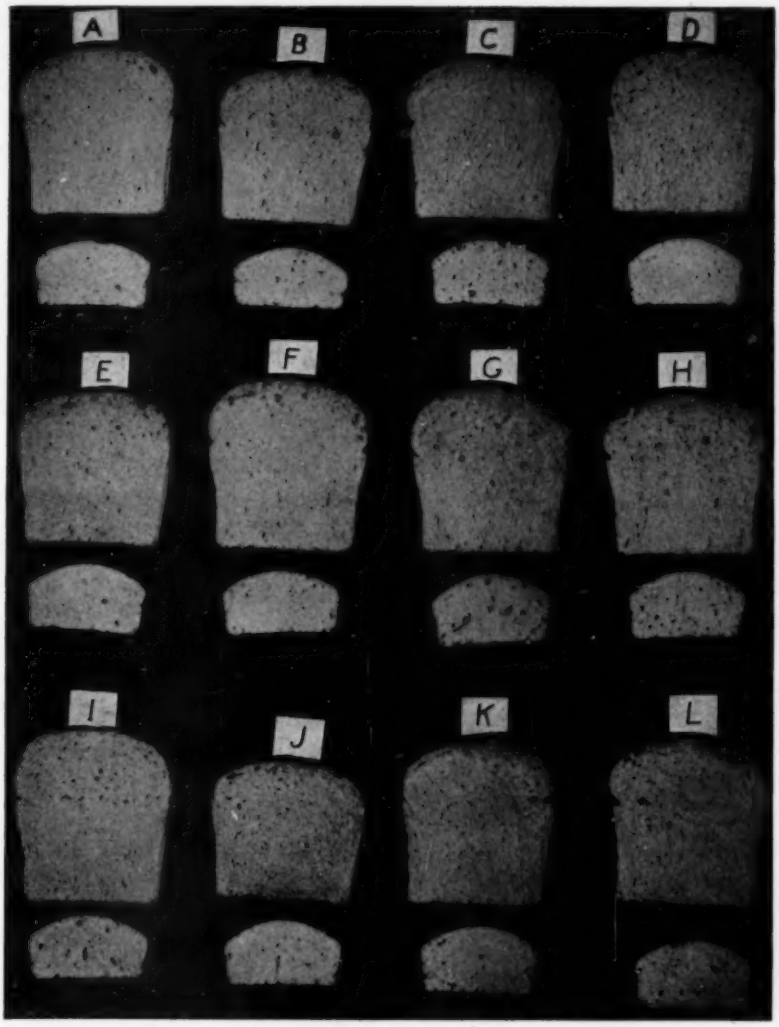


Fig. 2. Photograph of bread and cake made from the different wheat samples.

score was acquired by assigning numerical values to the important internal and external loaf characteristics. The items considered on the basis of 100 for a perfect score were volume, shape, bloom, break, color, grain, and texture.

The doughs all had typical soft-wheat mellowness and possessed, to only a slight degree, those dough properties which are characteristics for good bread production. The two white wheat samples had the weaker and poorer dough quality from the standpoint of bread production. These two wheats also had the least absorption and produced the smallest loaf volumes of the entire series. There were sufficient differences between the external and internal features of the different loaves in most instances to justify a distinguishing score. It is, however, definitely more difficult to differentiate between different soft-wheat flours by the bread-baking test than between unlike hard wheat flours.

Biscuit tests: The biscuit tests were conducted on a portion of the same bleached flour that was used for the bread-baking test. The baking technique followed was the same as that outlined by the Cincinnati Section, A. A. C. C. Research Committee (Schwain 1940). The formula was as follows: 125 g flour (15% moisture basis), 1.88 g soda, 2.34 g mono-calcium phosphate, 2.19 g salt, and 20.0 g shortening. The experience of the Cincinnati Section Research Committee indicated that the foregoing shortening level is near the optimum for soft-wheat-flour biscuit testing purposes. Preliminary bakes were used to determine the optimum absorption. The biscuits were scored by evaluating the various items enumerated by McKim and Moss (1939) and the numerical score assigned according to the A. A. C. C. score card as follows: grain 10, tenderness 10, flavor 20, crumb color 20, volume 40.

The volume and score of the biscuits baked from the various samples are recorded in Table II.

Cake tests: It is generally agreed that for the best cake results a flour should possess the following attributes: low moisture content, fine granulation, and a pH between 5.2 and 5.4. Before the cake-baking tests were started, the samples of flour were made to conform to the previously mentioned conditions. Portions of the original unbleached flour which had been milled on a Buhler mill were further reduced by use of the mill described by Libby and Shellenberger (1938) until 75% bolted through a 16XX silk. The overs and throughs were combined and rebolted through a 9XX to insure thorough remixing. No portion of each original sample was discarded.

The flour thus obtained was bleached with benzoyl peroxide and brought to a pH of approximately 5.2 by the use of chlorine. The actual pH range was found to vary from 5.19 to 5.34. The flour samples were all dried to the uniform moisture level of 11.8%.

The A. A. C. C. cake flour baking test was used and the cakes were scored according to the method suggested in *Cereal Laboratory Methods*

(1935). The volumes and scores are recorded in Table II, and in Figure 2 the cakes are compared with the bread baked from the same flour.

Cooky tests: Unbleached flour was used in the cooky test. The sugar cooky formula adopted was that recommended by Alexander (1933) as modified by Fifield, Bode, and Bayles (1936). It was necessary to reduce the absorption slightly in order to obtain a workable dough from these very soft wheat flours.

The cookies made from the various flours were remarkably similar, and it was very difficult to find distinguishing characteristics for grading purposes. Attempts to assign a numerical score to items such as symmetry, color, top grain, appearance, and crumb grain were abandoned. It was fairly obvious, however, that the two white wheat flours produced excellent cookies. In Table II the average thickness (T) and the average diameter (W) of the cookies are recorded and factor W/T is computed. The ratio of diameter to thickness is an index to the spread of cooky doughs, and consequently is an important criterion of quality. The cookies baked from white wheat flour had good spread.

Pie test: Unbleached flour was used in the pie-crust test. Bleached flour frequently produces pale pie crusts which are considered less desirable than when the color is slightly brownish. For this study the pie crust formula and method were those recommended by Kress (1932, 1935), except that these flours required less absorption than those investigated by Kress.

The pies were graded 24 hours after removal from the oven, and numerical values were assigned to the following qualities: dryness, color, tenderness, flakiness, and form. The relative score of the various pies is indicated in Table II.

Discussion of Results

A study of Table II indicates the difficulties and complications encountered in an attempt to evaluate soft wheat by test baking. There appears to be no very definite relationship between the various baking procedures. For example, the lack of conformity between the volumes of the bread and cake and the scores is evident in Figure 2 and Table II. One noticeable feature is that the two white wheat flours, namely E and J, are deficient in volume according to the bread-baking test, but are satisfactory in this respect when used in cakes. There is frequently an inverse relationship between bread and cake volumes obtained from the same flour, but there are many exceptions to this rule. Considered entirely from the standpoint of differentiating between soft-wheat flours of nearly the same quality, the bread-baking

test is preferable to the cake-baking test. The distinguishing characteristics of bread are generally more pronounced than the corresponding qualities of cake. Another item of no small consequence is the greater ease with which the bread-baking test can be carried out in comparison with the ease in conducting the cake-baking test.

There are a number of ways in which the bread-baking test can be used advantageously to predict the cake-baking quality of flour. When a cake flour has been evaluated by actual cake production, it is usually possible to establish by comparative bread-baking tests a series of dough and loaf characteristics which can be regarded as typical for that type of cake flour. The standards thus created can be used with convenience and reliability.

The fact that all samples except sample *B* gave fairly consistent results by the cake-baking test might justly raise a question concerning the value of the cake-baking procedure used. A baking test is of greatly diminished value if it will not differentiate between flour samples which have been milled from wheat known to be of different genetic make-up and grown under diverse climatic and soil conditions. It is not sufficient that the baking test detect poor results when bread flours or other unadapted flours are used in cakes. More convenient means are available for this purpose. A cake-flour test, to be of value, must be able to differentiate between flours which are all reasonably adapted to that purpose. Results of this series of tests, as well as other observations, lead to the conclusion that our present cake-baking test will not satisfactorily meet the foregoing condition. It may be that because of the ratio of other ingredients to flour in the cake-baking test, it can never be successfully adapted for the purpose of differentiating between flours of approximately the same quality.

The biscuit-baking test appears to classify flours entirely independently of their suitability for either bread or cake. The biscuit test was not especially critical and did not differentiate adequately between flours of the same general character.

The pie-crust baking test can also be included in the same category. The pie crusts from the twelve samples were very similar in most respects, and consequently the scoring was difficult and subject to considerable differences of opinion. One point worthy of special emphasis is the parallelism between the pie-crust score and the spread of cooky doughs as indicated by the factor W/T previously discussed. It is generally agreed that the greater the quotient obtained by dividing the average diameter by the average thickness of the same cookies, the better the cooky. Indications are that the cooky test provides a good indication of the desirability of a flour for pie crust and other pastries where spread and shortness are concerned. The

disadvantages of both the cooky and pie tests are the time-consuming operations required to perform the tests. It appears, however, that neither the bread, cake, nor biscuit tests can be used satisfactorily as a basis for predicting the suitability of a flour for doughs that require spread and shortness.

The present study also appears to indicate that the viscosity test is not closely associated with the various baking tests. Reference to Table I shows that the viscosity of the flour samples varied from 96° to 32° MacMichael. This viscosity range should correspond to a gradation in baking value, but as indicated by the scores recorded in Table II for the various baking tests, no very definite relationship exists.

Bayfield (1934, 1935) and Bayfield and Shiple (1937) made extensive studies of the methods of evaluating experimentally milled soft-wheat flours, and the viscosity test received considerable attention. As a measure of protein quality, it proved to be a promising but not entirely conclusive indication of flour strength. The viscosity test is extremely useful for differentiating between flours of different quality, but it cannot be used successfully without supplementary information to predict baking quality. Admittedly the baking methods used in this investigation have been submitted to a severe test. Possibly there is no justification for expecting baking tests to provide unmistakable evidence of dissimilarity between soft-wheat flours of nearly identical type. Also, in a comparison of methods of evaluating flours, consideration must be given to the fact that the interpretations of a test may perhaps be subject to as much criticism as is the method. Methods for interpreting the results of baking tests on soft wheat should continue to receive adequate consideration.

Cracker Sponge Studies

One of the unfortunate shortcomings of our soft-wheat testing techniques is lack of an informative baking test for cracker sponge flour. Attempts to bake certain types of crackers in the laboratory have never been entirely successful. It is possible to relate information obtained from the bread-baking test to the known suitability of the flour for cracker sponge doughs as determined by commercial use. This approach is of limited value and obviously cannot be very helpful to the plant breeder engaged in developing new wheat varieties that need to be tested for adaptability as cracker sponge flour types.

In an endeavor to follow the physical changes which cracker doughs undergo during extended fermentation, use was made of the micro recording dough mixer. The procedure was as follows: flour-water doughs containing 0.3% yeast and 50% absorption were mixed for 1½ minutes in the Hobart-Swanson mixer. Fifty-two grams of

each of these doughs were placed in the mixing bowl of the micro recording dough mixer and the curves obtained. In Figure 3 the upper curves are the ones obtained at the initial stage.

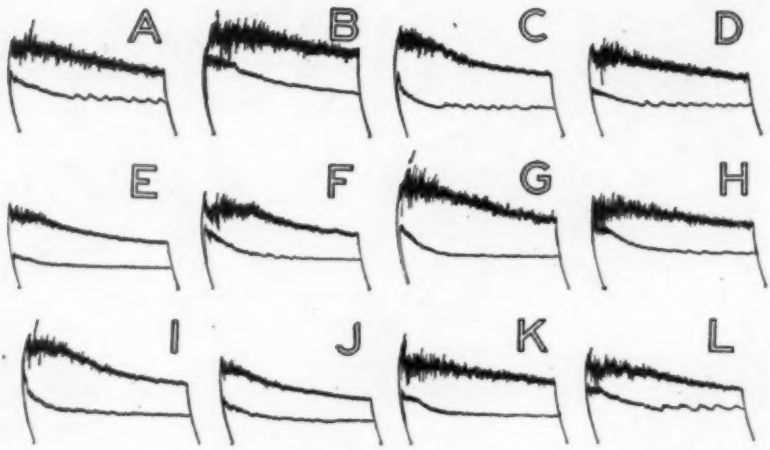


Fig. 3. Micro recording dough mixer curves of cracker sponge doughs before and after 20-hour fermentation period. Upper curves show initial stage.

The remaining portions of the large doughs were placed in a fermentation cabinet at 86°F and allowed to remain undisturbed for 20 hours. Fifty-two grams of the doughs were again weighed into the recording dough-mixer bowl and the curves recorded directly below the corresponding curve which was made when the doughs were first set. This is illustrated in Figure 3. The same damping adjustment was used in each instance.

These curves indicate very strikingly the different degrees of modification that aging brings about in the doughs. This is the result of a number of factors, such as the reorganization of the gluten strands, the effect of accumulated H-ions upon the dough proteins, and the partial proteolysis caused by the enzymes at work on the system. Landis (1935) has recommended the use of dough-mixing studies as an index of proteolytic activity. More recently Swanson (1940) has studied the effect of enzymes on curve characteristics and found, after a period of dough autolysis, that the curves were very similar to those produced by the addition of proteases. It is possible that with additional study the changes occurring in the physical condition of the dough, as indicated by mixing curves, may be correlated with the suitability of the flour for cracker dough production. Sample *A*, Figure 3, is an example of the curves from a satisfactory cracker sponge flour. The thixoliopic condition of the doughs is indicated by

the decreased mobility after a period of quiescence and the subsequent drop in shearing force as the dough becomes a sol. The curves of sample *B*, for example, are noticeably different from those of sample *A*. Sample *E* represents another type of curve where even less strength is evidenced.

Summary and Conclusions

This investigation had as its objective the determination of the relationship between the various baking tests that can be applied to soft wheat. Information was also sought on the degree of differentiation between samples which the various baking tests are able to disclose.

Twelve samples of soft wheat grown under different environmental conditions and differing genetically were obtained. These samples were experimentally milled and several portions of the flour were especially treated to improve their baking response for specific tests. Comparative bread, cake, biscuit, cooky, and pie flour baking tests were conducted and the products evaluated by careful scoring.

Considering the subject entirely from the standpoint of differentiation between flours, the bread-baking test has greater merit than any of the others. This test, however, leaves much to be desired. The limitations of the bread baking test, when applied to soft wheat, have been previously discussed by Bayfield and Shiple (1933) and Bayfield (1934). It is true that the test is not perfectly adapted to soft wheat flour studies, but, as indicated by Shellenberger (1940), it is possible to obtain much valuable information by its use.

Except for the parallelism between the cooky and pie crust tests, there is no apparent relationship between the baking tests. Neither the volumes nor the scores of the bread, cake, and biscuit tests correspond sufficiently to warrant prediction of the baking results of a flour for other than one purpose. Without supplementary evidence or experience applicable to the flour under investigation, it is unwise to attempt the precise evaluation of a flour for purposes other than that indicated by the particular baking test used.

Present baking methods are satisfactory for the broader classification of wheat flours into categories such as bakery, family, and pastry types. The vital need at present is for baking and interpretive methods that can be used with reasonable ease and reliability to predict the relative value of a soft-wheat flour within the broader classification. Our present baking-test methods apparently indicate the potentialities of a flour for only one purpose, thus necessitating the tedious task of conducting separate baking tests for the complete evaluation of the flour.

The micro recording dough mixer is useful for indicating differences in the dough characteristics of soft-wheat flours. There is evidence that this instrument may be useful in helping to characterize cracker sponge flour.

Literature Cited

Alexander, G. L.
1933 The results of bleaching Michigan soft winter wheat cake flours by the Brabender electric bleaching apparatus. Cereal Chem. **10**: 623–626.

American Association of Cereal Chemists
1935 Cereal laboratory methods (3rd ed.).

Bailey, C. H.
1940 Physical tests of flour quality. Wheat Studies of the Food Research Institute, Vol. 16, No. 6.

Bayfield, E. G.
1934 Soft winter wheat studies. II. Evaluating experimentally milled flours with the aid of viscosity, fermentation, and baking tests. Cereal Chem. **11**: 121–140.
1935 Soft winter wheat studies. III. The effect of some factors influencing viscosity and protein. Cereal Chem. **12**: 1–16.

—— and Shiple, V.
1933 Soft winter wheat studies. I. The suitability of the A. A. C. C. basic baking procedure for the determination of strength. Cereal Chem. **10**: 140–148.
1937 Soft winter wheat studies. V. Evaluating the quality and the strength of some varieties. Cereal Chem. **14**: 551–577.

Blish, M. J., and Sandstedt, R. M.
1933 An improved method for the estimation of flour diastatic value. Cereal Chem. **10**: 189–202.

Fifield, C. C., Bode, C. E., and Bayles, B. B.
1936 Chemical, milling, and baking results for wheat varieties grown in the cooperative varietal experiments in the Western Region in 1934. U. S. Department of Agriculture, Bureau of Plant Industry. Mimeographed report.

Kress, C. B., Chairman
1932 Report of the subcommittee on pie-flour tests. Cereal Chem. **9**: 411–413.
1935 Report of the 1933–34 pie-flour committee. Cereal Chem. **12**: 158–160.

Landis, Q.
1935 An index of proteolytic activity by use of the farinograph. Cereal Chem. **12**: 25–33.

—— and Frey, C. N.
1933 Diastatic supplements for the A. A. C. C. baking test. Cereal Chem. **10**: 588–592.

—— —— and McHugh, S. A.
1935 Diastatic supplements for the A. A. C. C. baking test. Cereal Chem. **12**: 302–304.

Libby, J. J., and Shellenberger, J. A.
1938 Unique experimental mill. The Northwestern Miller (Production Number) Vol. 193, No. 7.

McKim, Elizabeth, and Moss, H. V.
1939 Study of definition and evaluation of various items on score card. Cereal Chem. **16**: 117–126.

Schwain, F. R.
1940 Report of the 1939–1940 research committee, Cincinnati section A. A. C. C. Mimeographed report.

Shellenberger, J. A.
1938 X-ray photographs as a means of determining when a dough is adequately mixed. Cereal Chem. **15**: 197–200.
1940a Bread baking test suggested for pastry flours. Food Industries **12**: 52–54.
1940b A physical dough testing method applied to flour mill stream analyses. The Northwestern Miller (Production Number) Vol. 204, No. 5.

Sherwood, R. C., and Bailey, C. H.
1926 Control of diastatic activity in wheat flour. II. Experiments with flour milled on a commercial scale. Cereal Chem. **3**: 163–182.
Swanson, C. O.
1940 Factors which influence the physical properties of dough. I. Effects of autolysis on the characteristics of dough mixer curves. Cereal Chem. **17**: 689–700.
Ziegler, E.
1938 The use of a purifier in experimental milling. Cereal Chem. **15**: 663–671.

SOME EFFECTS OF REWORKING FERMENTING DOUGHS

E. N. Frank

International Milling Co., Minneapolis, Minnesota

(Read at the Annual Meeting, May 1941)

Freilich and Frey (1938) have demonstrated that remixing of fermented doughs will overcome a certain type of tightness or buckiness. They worked with straight doughs and for their purpose induced this condition by the addition of amounts of potassium bromate greatly in excess of those ordinarily employed in baking. Their work suggested that the same principle of treatment might be effective in improving the action of doughs which display natural buckiness.

Materials Used

Samples of hard wheats which produce doughs with natural buckiness are not infrequently encountered but the frequency varies from crop to crop.

Our laboratory was one of fifteen which collaborated in the testing of the 1940 crop spring wheat samples produced for the Northwest Crop Improvement Association. When the usual testing had been completed, we still had on hand a residual portion of the experimentally milled flour from each of the 24 wheats. Notations on our baking records indicated a wide range in the degrees of buckiness of the respective doughs. Two composites were therefore formed using eight of the flours. The four which had shown the most buckiness were blended to form a flour to which we gave the designation "bucky" and the four with least evidence of this characteristic to form the one designated as "nonbucky." The former showed by analysis 0.42% ash and 16.60% protein, and the latter 0.41% ash and 14.90% protein.

Experimental

The formula and procedure for the bakings to be described, unless otherwise indicated, were essentially those for the standard A. A. C. C. test. The doughs contained the following ingredients in addition to

those specified for the standard test: 1½% sugar (additional to the specified 2½%), 0.1% ammonium carbonate, 2% lard, and 0.5% malted wheat flour. Doughs were mixed with a Hobart-Swanson machine and baked in the tall-form pans. All tests were made in sextuplicate. The two composite flours were first subjected to a simple mixing differential test. Results of this baking are shown in Table I.

TABLE I

EFFECT OF INITIAL MIXING PERIOD UPON LOAF VOLUME

Sample	Average loaf volume		Response
	2 min mixing (Schedule A)	3 min mixing (Schedule B)	
	cc	*cc*	*cc*
Bucky	810	693	−117
Nonbucky	847	879	+32

There was no marked difference of external appearance between the loaves from doughs which had been mixed for two minutes but there was a distinct difference in the three-minute loaves. Here the loaves from the bucky flour had a very rough break and shred, while those from the nonbucky one were externally smooth.

The two flours were next submitted to a series of baking tests in which remixing at various stages of fermentation was employed. The mixing schedules are shown in Table II. Average loaf-volume figures obtained are given in Table III. The data appearing in Tables I and III are shown graphically in Figure 1.

TABLE II

SCHEDULES OF REMIXING

Schedule	Initial mixing	Remixed in place of first punch	Remixed in place of second punch	Total mixing
	min	*min*	*min*	*min*
C	2½	½	0	3
D	2½	0	½	3
E	2	½	½	3

TABLE III

EFFECTS OF REMIXING UPON LOAF VOLUME

Sample	Schedule C	Schedule D	Schedule E
	cc	*cc*	*cc*
Bucky	835	888	933
Nonbucky	826	775	737

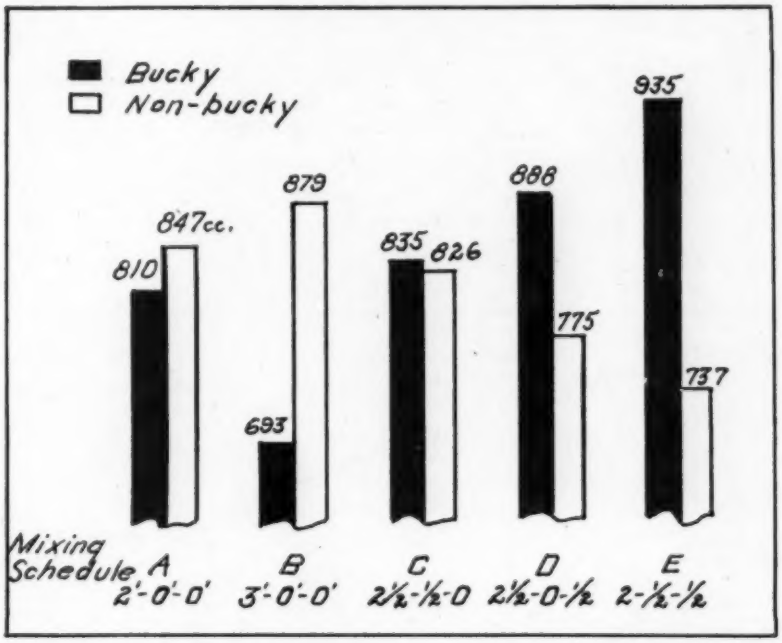


Fig. 1. Effect of remixing of doughs on loaf volume.

It is at once evident that every change in the mixing treatment which benefited the doughs of one flour handicapped those of the other. Deferment of a portion of the mixing treatment was of distinct benefit to the doughs from the bucky sample. For this flour the third minute of the initial mixing period was detrimental, but the same amount of mixing deferred to points of time when the doughs were in later stages of development was beneficial to loaf volume.

Further bakings were made using the three-minute initial mixing period and reworking the partially fermented doughs in a different manner. This was accomplished by passing them repeatedly through sheeting rolls to give the effect of braking. Preliminary experiments showed that a convenient and satisfactory stage at which to apply this treatment was at the time and in the place of the usual second punch. The number of passages through the rolls was increased by stages from 0 to 12 in multiples of 4. The effects upon loaf volume are indicated in Table IV and Figure 2.

Four passages through the brake caused an increase in loaf volume for both samples relative to their controls but with further increase of the braking effect, the volume of loaves from the bucky sample increased while that of the nonbucky one decreased. This is in general agreement with the effects of reworking by remixing.

TABLE IV

EFFECT OF BRAKING UPON LOAF VOLUME

Sample	Number of times through the brake			
	0	4	8	12
	cc	*cc*	*cc*	*cc*
Bucky	688	726	758	773
Nonbucky	841	855	800	749

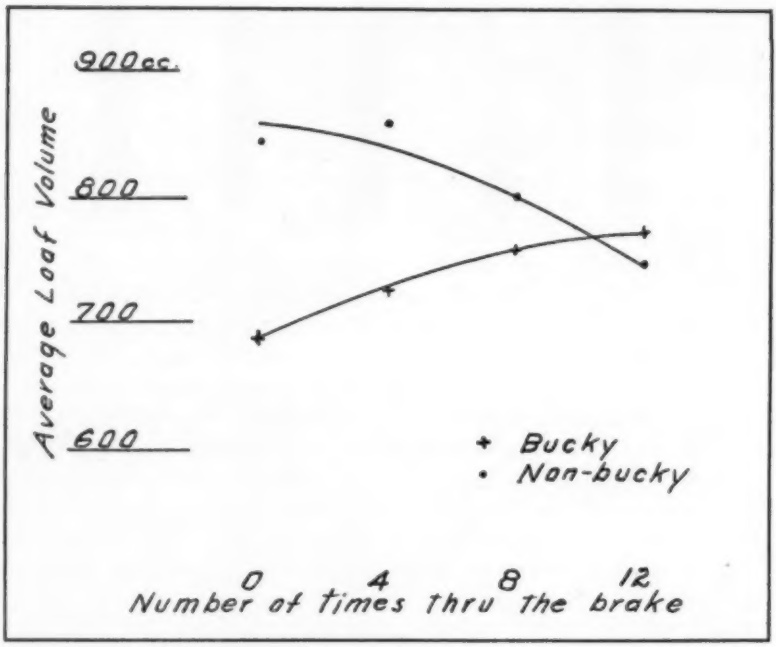


Fig. 2. Effect of braking of doughs on loaf volume.

The break and fill-in of the loaves from the bucky sample became progressively smoother with increased application of the braking treatment, while those of the nonbucky one were smooth throughout the entire series.

Discussion

Sufficient experimental work to establish the immediate cause of natural buckiness has not been done. It appears to be due to a partial coagulation of the gluten proteins. It is favored in its development by increased oxidation due to prolonged initial mixing in contact with air and by increased hydrogen-ion concentration as fermentation proceeds.

It is retarded by mechanical reworking of the fermenting dough which probably prevents the formation of, or causes the destruction of, some type of association of particles. It is permanently removed by reworking the fully fermented dough.

The phenomenon may be due to thixotropism of the starch component although, if so, the observed effects of reworking should have been highly reversible. Markley (1937) has stated that: "A thixotropic system is one which is a gel when quiescent, but which becomes a sol upon the application of a shearing force, but again becomes a gel when allowed to return to the quiescent stage. . . . This process can be repeated many times."

The cause of natural buckiness in doughs from certain flours is suggested as an interesting subject for further research.

Summary

When judged by the results of a fixed straight-dough baking procedure employing a severe initial mixing treatment, there was a wide range in the degrees of buckiness of the respective doughs from a group of spring wheats grown in a single crop year.

Variations in the schedule and severity of mixing and of the mechanical working of fermenting doughs significantly affected their relative conditions of buckiness at the subsequent molding stage.

Under the conditions employed, remixing produced a larger effect than braking.

For a sample with an inherent tendency toward buckiness, extension of the initial period of mixing accentuated this characteristic; but deferment of a portion of the mixing treatment or more thorough reworking of the doughs at later stages of development reduced it.

Literature Cited

Freilich, J., and Frey, C. N.
1938 Dough oxidation and mixing studies. II. Cereal Chem. **16**: 495–502.
Markley, M. C.
1937 Colloidal behavior of flour doughs. I. Cereal Chem. **14**: 434–436.

INTERPRETING EXPERIMENTAL MILLING DATA FROM A COMMERCIAL ASPECT

PERIE RUMOLD

Standard Milling Co., Kansas City, Kansas

(Read at the Annual Meeting, May 1941)

Experimental milling is recognized as an essential operation in many cereal laboratories. The purpose of the miniature mills is to provide flour for the experimental baker and the analyst for comparative tests with known standard varieties and standard commercial blends.

Experimental milling work is usually directed toward one of two main objectives: first, aiding plant breeders and agronomists in selecting new wheat varieties and, second, providing the commercial miller with advance milling data and the mill chemists with sufficient flour for preliminary test baking and analytical investigation. This information is valuable to the wheat purchasing department in selecting wheats that are satisfactory for future mill blends.

This discussion will deal principally with procedure and interpretation from a commercial and industrial viewpoint. The system used by some commercial mill laboratories will be outlined for the purpose of indicating the method of correlating the experimental mill results with actual commercial production.

The routine used in a mill laboratory will depend largely upon the ratio of wheat in storage to the output of the commercial mill. A system of experimental milling used where four or five months' supply of wheat is available may prove unsatisfactory where only a few weeks' supply of wheat is at one's disposal.

Milling tests should be made regularly, and it is very important to thoroughly clean out the mill before starting a day's run. One or two samples should be milled at the beginning and the results disregarded. Repeated tests in our laboratory indicate that dependable results are obtained only when beginning with the third sample. When time and help are limiting factors I think that far more reliable results can be obtained by milling four or five samples at a time than by milling one or two samples a day on several different days.

Some laboratories use the following method to determine the merits of new crops and to establish flour standards for the ensuing year. First each bin of wheat is selected according to protein, test weight, type of wheat, and point of origin. Then the wheat is thoroughly blended in such a manner that a sample of wheat taken at any time will be representative of the entire lot. Each bin is now

given a lot number and each lot is milled and the flour analyzed and baked. Various laboratories make from one to four millings on each lot of wheat. Additional milling tests can be made whenever the wheat is turned, to show the effect of storage and aging upon the milling and baking qualities of the wheat stocks.

Upon completion of this type of work one can make up blends from the various lots that will compare with those that have been used during the previous crop. After these combinations have been tested one can adopt a standard for each mix that is to be used as the basis of comparison between the old and new crop. This will determine the potentialities of the lots of wheat and wheat mixtures so that standards for the new crop can be established.

In designing an experimental milling system we have found that it is impractical to duplicate commercial milling results. Since the primary purpose of experimental milling work is to determine the comparative values of the different types of wheat a set procedure has been adopted.

One-thousand-gram samples are weighed and passed over a combination cleaner and scourer and the screenings are weighed to determine the cleaning loss. Then water is added and the wheat stirred until the moisture is uniformly distributed throughout the sample. The amount of water added depends on the moisture content and the type of wheat to be milled. The wheat is tempered in the late afternoon, placed in a closed metal container, and allowed to stand overnight.

The following day the tempered wheat is passed over the break rolls. Four or five breaks are used, depending on the cleanup desired. Two stands of break rolls are used with the following corrugations: one stand, fast roll, 12 corrugations to the inch, slow roll 20 corrugations to the inch and running dull to dull. The other stand has 24 corrugations to the inch on both rolls, running dull to dull. All corrugations are standard Dawson cut. Practically all of the endosperm is removed in the first three breaks and only a small amount of fine and inferior middlings are obtained from the fourth and fifth breaks.

The sizings and middlings stocks are now carefully reduced through a 64 GG sieve. At this stage as little flour is produced as possible. When the coarse middlings have been reduced through the 64 GG sieve the stock is sifted to reclassify the middlings and remove the flour that has been produced thus far. The classified stocks are now ground into flour, which takes from three to four reductions.

This system will yield approximately 625 grams of flour without grinding any stocks that contain large amounts of bran particles.

To increase the yield of flour we use the finer corrugated rolls on the last two breaks and further reduce the stocks not used in making the above-mentioned 625 grams of flour.

The sieve arrangement used on the breaks and coarse middlings reductions are, from top to bottom:

16 W
38 GG
64 GG
Blank

and on the reclassification and final reductions the sieves are stacked top to bottom:

Hard wheat	*Soft wheat*
64 GG	64 GG
8 XX	11 XX
11 XX	13 XX
Blank	Blank

The operator attempts to break the wheat in such a manner as to obtain a definite amount of products on each break, but there is apt to be a variation due to the type of wheat and the setting of the rolls. This depends upon the condition of the wheat before milling, atmospheric conditions, the type of wheat, and the judgment of the operator. The same procedure is used in reducing the middlings, and the cleanup depends entirely upon the skill of the operator.

Inasmuch as it is impossible to condition each type of wheat separately in the present-day arrangement of our commercial units, wheat or types of wheat that are to be mixed into a common blend are tempered or conditioned in the same manner. Without a definite system of conditioning one can lose sight of the milling characteristics if every sample is to be milled in a special manner to suit that particular lot of wheat.

The results obtained by this set procedure can be correlated with the results achieved in the commercial mill. Determining the ash content of the flour can be used to good advantage in deciding the best method of milling and to what degree the wheat must be tempered to obtain flour with the lowest ash content.

Assuming that one has equipped the laboratory with a satisfactory experimental mill and has a well established system of milling, it is necessary to provide a well trained and capable technician in order to obtain results that are reliable and comprehensive. Sometimes little thought is given to experimental mill work and its relation to commercial production. If insufficient capital is provided for proper facilities and personnel one cannot expect to derive maximum benefits from this phase of laboratory work.

A good technician should be able to judge milling values by observing such characteristics as potential extractions and flour yields, bran thickness, ability of the bran to hold together during the breaking operation, nature of middlings reduction, the character of the ground stock, and the time required for each sifting. All of these factors are very important to the mill superintendent and if properly interpreted and disclosed in an understandable manner they can serve as an important guide to the operative miller in the manufacture of a satisfactory and uniform flour. Close observation of the scalps from all the reductions in addition to the flour obtained will enable one to estimate the approximate yield of flour expected from a given lot of wheat.

It is very important to detect musty and sour odors, mold, heat and other forms of damage, foreign material of all kinds, cracked kernels, shriveled grain, and presence of smut and other abnormalities that are present in wheat samples from time to time. Occasionally a small amount of contaminated wheat that would escape the notice of an inexperienced operator could cause sufficient trouble to warrant the extra expense of employing a dependable operator. Frequent talks with the experimental miller tend to encourage efficient operation and help him to realize the importance of his work. I think that the quality of this type of work could be improved by including it as an educational and training course for the personnel in the wheat handling or wheat purchasing department.

I am sure most millers will agree that the results obtained from any experimental or commercial mill are greatly influenced by atmospheric conditions prevailing in the room during the milling process. At present very few experimental or commercial mills are equipped to control these conditions. The significance of uniform milling conditions is being recognized as one of the most important factors in both experimental and commercial mill work. From time to time this is brought to our attention by the invention and installation of equipment intended to artificially control or condition the air of flour mills.

The fact that experimental milling results vary with temperature and humidity has been emphasized by the work of Bayfield in recent years. This is further substantiated by experimental investigations in commercial mill laboratories. If milling results are to be strictly comparable they should be conducted under identical atmospheric conditions. There is a fairly close agreement among experimental millers that best milling results are obtained with a temperature of 78°–82°F and a relative humidity of 60%–65%, and if these conditions are maintained milling results can be duplicated from day to day. It is well to keep in mind that the atmospheric conditions must be

constant if one wishes to make direct comparisons between commercial and experimental mill results. Millers and mill chemists agree that it is advisable to make comparisons with the same commercial mill unit, as it has been observed that results are not duplicated on different milling units.

It is readily possible to establish a basis for correlating experimental mill work with the operations of the commercial mill. We have found, however, that there will be occasional variations in the commercial mill that are not reflected on the same wheat blends when milled on the experimental mill. Therefore it is essential to check experimental mill results with the standard for any particular mix that has been established for experimental mill work. This procedure has been found acceptable to our production department, and its use has enabled us to inform that department of factors that are instrumental in maintaining efficient operation.

EXPERIMENTAL DURUM MILLING AND PROCESSING EQUIPMENT, WITH FURTHER QUALITY STUDIES ON NORTH DAKOTA DURUM WHEATS[1]

R. H. HARRIS and L. D. SIBBITT

North Dakota Agricultural Experiment Station, Fargo, North Dakota

(Received for publication August 27, 1941)

Harris and Knowles (1940) published data obtained from quality studies conducted upon the 1938 crop of North Dakota durum wheat. The wheats had been milled and the semolina processed into macaroni at the Dominion Grain Research Laboratory at Winnipeg, Canada, because of lack of standard equipment at the North Dakota Station.[2] The apparatus needed to carry on investigations interpretable in terms of commercial practice has since been purchased, and is described in some detail in this report. The present paper also contains data derived from a continuation of preliminary work reported previously, and comprises material obtained from the milling and processing of wheats produced in the crop years 1939 and 1940. Cooking-quality studies on the macaroni are planned for a later date, and will be published in due course.

Fifield (1934) and Fifield, Smith, and Hayes (1937) have discussed experimental equipment and methods for the manufacture of macaroni products, and have published results obtained from durum wheats

[1] Published with the approval of the Director of the North Dakota Agricultural Experiment Station.
[2] L. D. Sibbitt, who was at that time experimental miller at the Dominion Grain Research Laboratory, was instrumental in performing the milling, processing, and quality evaluation of the macaroni.

grown in the hard red spring wheat region of the United States from 1932 to 1936. Binnington and Geddes (1936) described in detail experimental milling and processing apparatus for durum wheats, and presented a statistical basis for the evaluation of the results. Later Binnington and Geddes (1937) published data derived from a study of 34 samples of Canadian durum grown in 1934 and 1935. Significant differences in macaroni color and appearance were demonstrated among the samples. Further studies by Binnington and Geddes (1939) emphasized the point that macaroni quality cannot yet be predicted from any single analytical test applied to the wheat and that wheat carotene is valueless as an index of macaroni color, particularly for inter-varietal prediction.

Milling and Processing Equipment

The milling equipment consists of a two-stand Allis-Chalmers experimental mill, equipped with one bolter and a small-scale purifier. The mill is provided with 19th middlings cut rolls (6 × 6-inch), one stand fitted with No. 16 and the other with No. 24 corrugations, both sets with ¾-inch spiral and running dull to dull. A photograph of the mill is shown in Figure 1.

The purifier is a modified form of a Minneapolis commercial-type machine which was scaled down to laboratory dimensions by the removal of a screw conveyor in the base and the traveling brush attachment for cleaning the sieve. The sieve was also removed and a more suitable one (6 inches in working width) substituted to prevent portions of the sieve running bare when operating. The main sieve is divided into four portions consisting of sizes 50GG, 40GG, 34GG, and 28GG and is activated by a 3-horsepower electric motor. Three compartments were installed in the base of the purifier to receive the purified semolina and a suitable fan was housed at the rear top to furnish needed aspiration. The air current can be varied in intensity by levers placed outside the housing which regulate the size of the openings of the air channels from the fan chamber to the purifier proper. A dust collector was located in the basement of the building and the exhaust was piped to it. This machine is represented in Figure 2.

The macaroni processing apparatus consists of a mixer, kneader, and press mounted on a common table and driven by a 1½-horsepower motor. The set-up closely resembles the one described by Fifield (1934) and Binnington and Geddes (1936) and is shown in Figure 3. Limit switches stop the motor at either end of the press travel, and a small motor-driven propeller mounted on the jacket insures proper agitation of the oil bath surrounding the press chamber, which has a thermostatic temperature control.

The drying cabinet is modeled after the one described in detail by Binnington and Geddes (1936) with the exception that no refrigerating unit was included to aid in removing moisture from the air within the cabinet. A small vent in the system is opened gradually to permit the moisture-laden air to escape. The interior of the drier is lined with

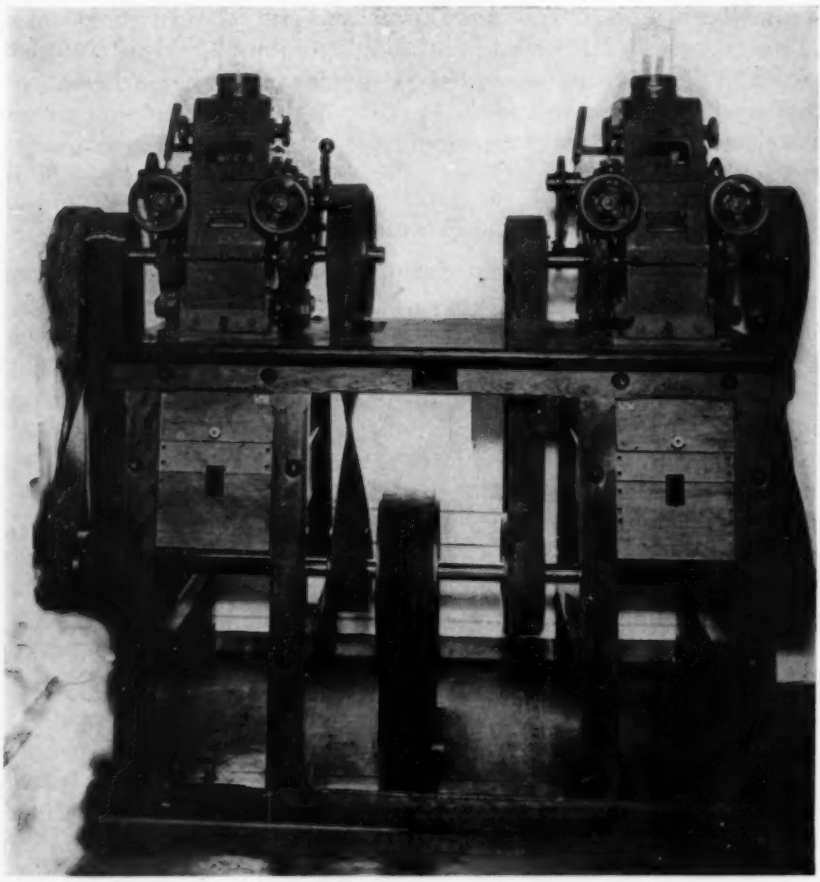


Fig. 1. Experimental durum wheat mill.

thin sheet copper and a suitable set of louvres with handles projecting outside the cabinet was installed at either end of the drying chamber.

A Fenwall thermoregulator is used to control the temperature through a 192-watt heater. The control bulb of this instrument, placed in the air-flow coming directly from the humidifying and heating chambers, proved very satisfactory for holding the temperature at a constant level during drying.

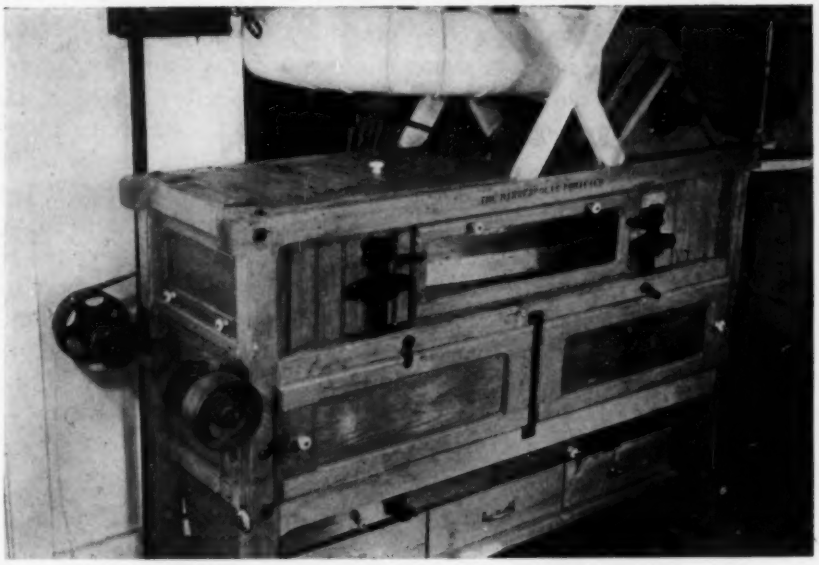


Fig. 2. Experimental purifier used in durum wheat milling.

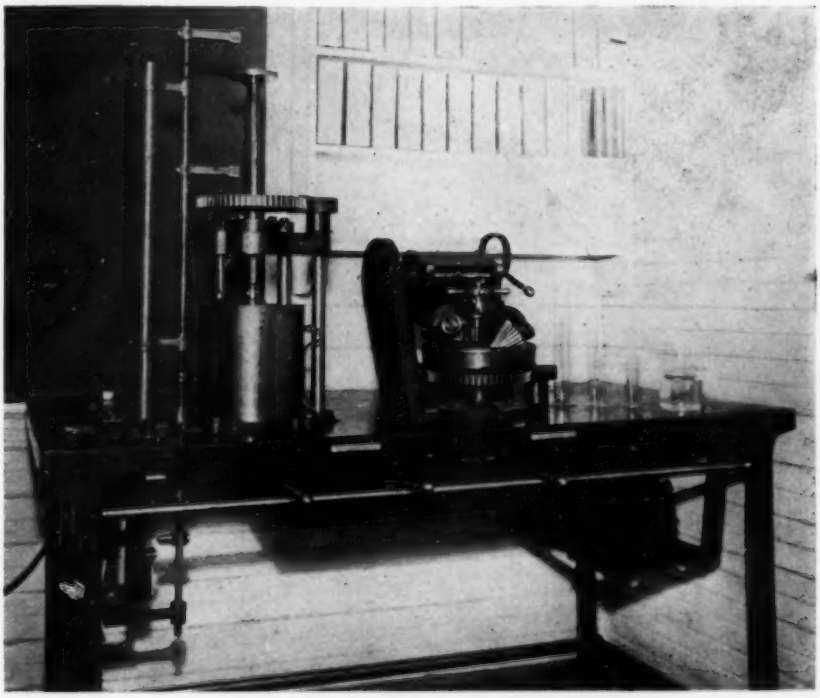


Fig. 3. Experimental macaroni processing unit.

A variable resistance boiler of the type described by Binnington and Geddes is employed as a source of humidity. A vacuum-tube relay is used to control the input of current to the boiler. This relay is connected to the wet-bulb pen of the Taylor temperature recorder, and functions by permitting the electric current to pass directly to the carbon electrodes of the boiler when it is necessary to raise the humidity of the cabinet. When the cabinet humidity has reached the desired point the current is shunted to the boiler through a resistance lamp bank. Under these conditions sufficient heat is generated by the reduced current to maintain the water in the boiler just below the boiling point. Suitable charts were constructed to control the relative humidity at the proper levels during the drying period. The drier is shown in Figure 4.

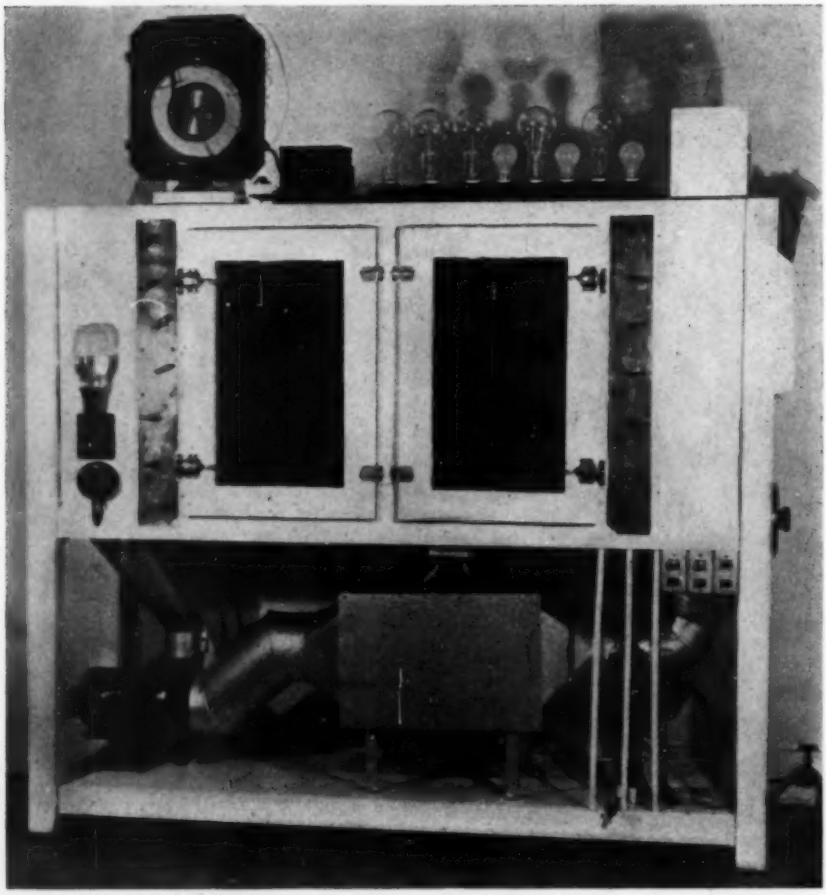


Fig. 4. Experimental drying cabinet with temperature and humidity control equipment. Relay and lamp resistance bank shown at top of drier.

Milling Technique

Cleaned wheat is used for the milling tests and the milling samples are weighed to give 3,000 g of wheat on a 13.5% moisture basis. Sixteen hours before milling, sufficient water is added to bring the moisture content to 13.5%. At the end of the conditioning period the sample is scoured and the moisture raised to 15.5% 90 minutes prior to milling. The mill laboratory temperature is held at approximately 70°F and the relative humidity at about 60%. The milling is conducted according to the flow sheet shown in Figure 5. Ten breaks are employed and all

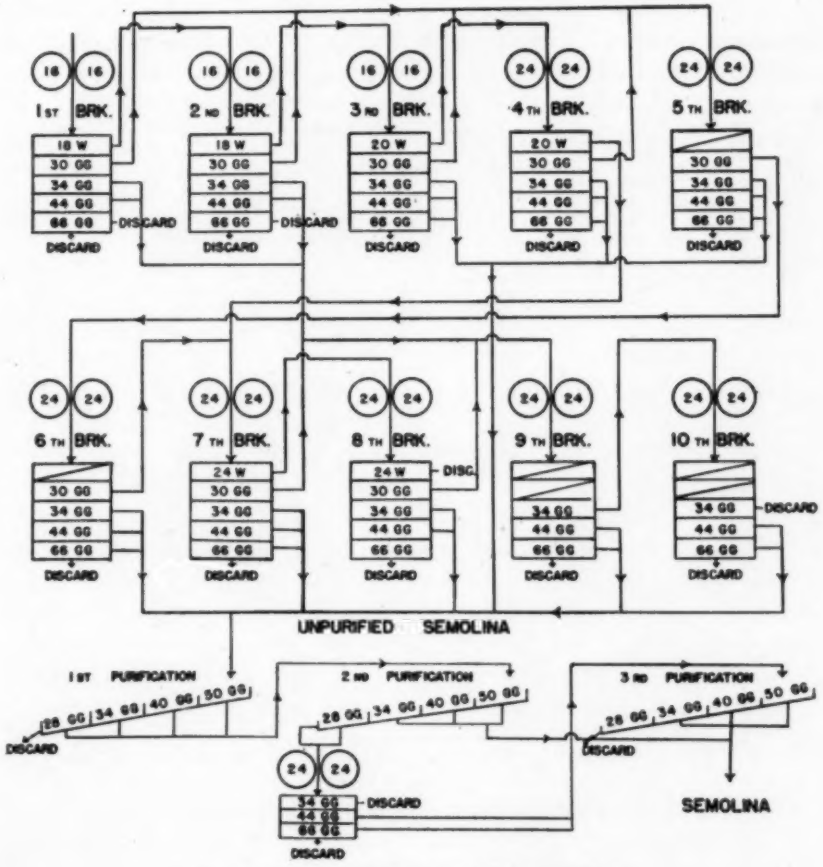


Fig. 5. Experimental flow sheet for durum milling.

stock after the second break, passing through a No. 30GG and retained on a No. 66GG, is bulked together and weighed as unpurified semolina. This material is then passed through a purifier in a thin stream with a light air flow. The semolina passing through the sieves of the purifier

is bulked together and passed through a second time, the tailings being discarded. In this second purification the maximum air flow obtainable is used. The throughs from Nos. 50, 40, and 34GG are composited. The tailings and the throughs from No. 28GG are reground and bolted, and the material remaining on Nos. 44 and 66GG is given a third purification. The throughs from Nos. 50, 40, and 34GG are thoroughly mixed with the material taken off in the second purification and weighed as purified semolina.

Processing Technique

The major operations included in macaroni processing are mixing, kneading, pressing, fanning, and drying. The laboratory is maintained at 55% to 60% relative humidity while the processing is being

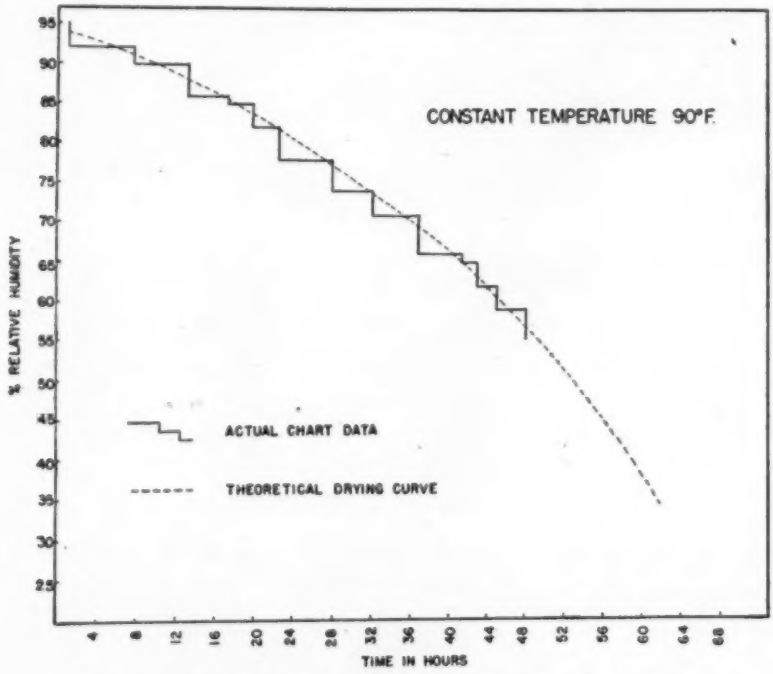


Fig. 6. Time-humidity gradient used in the experimental drying of macaroni.

done. The press temperature is held at 92°F. A special macaroni die is used which is substantially thicker than the customary experimental dies and has its center rod held firmly by three knife-edge supports instead of one, as is usual in experimental dies. This arrangement prevents possible displacement of the center rod during pressing with resultant variations in macaroni wall thickness. In processing, 600 g

of semolina on a 13.5% moisture basis is used. Sufficient water is added to form a stiff dough, the semolina and water are then mixed, and kneaded to optimum consistency. The dough is permitted to rest for ten minutes at press temperature before being pressed into macaroni. The 30-inch lengths of macaroni are suspended over wooden rods and surface-dried at room temperature in an air current from a fan. The material is then placed in the drying cabinet where it is sweated for a minimum period of one hour at 90°F and 95% relative humidity. The drying of the macaroni is performed in the cabinet, which is fitted with devices for accurately and automatically controlling the temperature and humidity.

The apparatus and methods employed closely resemble those used in commercial semolina and macaroni manufacture. Drying is done at a constant temperature and under a falling humidity gradient as represented in Figure 6. The visual color score of the macaroni was determined under a mercury fluorescent lamp.

Material

Thirty-two samples of durum wheat grown at Fargo and Langdon [3] were experimentally milled and the semolina processed by the equipment described, using the standardized techniques developed by Binnington and Geddes (1936). The moisture and protein contents of the wheat and semolina were determined, as well as the number of specks per ten square inches of semolina. It is somewhat unfortunate that only wheat of the 1940 crop was available at Langdon as the district is noted for high-quality durum production, but the quality suffered severely from damage caused by fungus attacks and other injurious factors which were favored by unfavorable weather conditions during July, August, and September, as pointed out by Harris and Sibbitt (1941). The results obtained on the Langdon wheats are markedly lower as a result of the effect of these conditions. Fargo lies southeast of the area of best-quality durum production for macaroni purposes, and the plots at this station escaped substantial damage in 1940.

Discussion of Data

In Table I are shown the description of the wheats with associated protein and milling data. A number of varieties were included in these series of wheats to cover fairly well the field of quality. Several of the varieties, such as Pentad, Golden Ball, and Monad, are well known to be unsuitable for the production of quality macaroni. Other varieties are under test with the view of introducing them for general production

[3] The varieties from this Station were grown in cooperation with the Division of Cereal Crops and Diseases, Bureau of Plant Industry, U. S. Department of Agriculture.

TABLE I

Wheat Description, Protein, and Milling Data on Durum Wheats Grown in 1939 and 1940

(Results arranged in order of increasing wheat protein content within stations)

Lab. No.	Variety	Test wt. per bu.	Unofficial grade	Kernel damage			Protein (N × 5.7) [1]		Semolina yield	
				Light	Heavy	Total	Wheat	Semolina	Unpurified	Purified
		lbs		%	%	%	%	%	%	%
				FARGO 1939						
39–643	Golden Ball	62.1	1 HAD[2]	—	—	—	15.6	13.3	65.7	43.0
646	Kubanka 314	63.1	1 HAD	—	—	—	15.4	13.7	65.4	43.3
644	Pentad	63.8	RD	—	—	—	14.3	12.1	65.7	43.1
649	Ld 9 × Min	64.1	1 HAD	—	—	—	14.3	13.0	65.0	45.6
648	Ld 34	63.7	1 HAD	—	—	—	14.2	12.8	66.2	45.3
647	Kubanka 74	63.6	1 HAD	—	—	—	13.9	12.6	65.9	44.4
641	Kubanka	63.8	1 HAD	—	—	—	13.8	12.4	65.3	43.3
640	Mindum	64.0	1 HAD	—	—	—	13.5	12.5	66.2	44.4
642	Monad	63.9	1 HAD	—	—	—	13.5	11.6	66.6	45.1
645	Kubanka 49	64.4	1 HAD	—	—	—	13.1	12.0	64.4	43.3
				FARGO 1940						
40–900	Ld 134	64.0	1 HAD	Some broken kernels			14.4	12.9	64.8	43.6
897	Ld 102	63.9	1 HAD	Some black point			14.2	12.7	65.8	43.3
894	Kubanka	63.9	1 HAD				14.1	13.1	66.5	47.7
898	Ld 104	64.6	1 HAD	Some black point			14.0	12.6	67.1	43.7
895	Pentad	64.0	RD	Some black point			13.9	12.5	65.9	44.9
899	Ld 111	64.5	1 HAD	Trace black point			13.9	12.2	65.0	42.5
896	Ld 34	64.5	1 HAD	Some black point			13.8	12.3	66.6	46.2
902	Kubanka 314	64.2	1 HAD				13.5	12.1	65.7	44.6
893	Mindum	63.8	1 HAD	Some black point			13.4	12.3	65.6	44.6
901	Kubanka 49	64.5	1 HAD				13.2	12.0	64.1	42.8
				LANGDON 1940						
40–876	Ld 101	60.8	4 HAD	3	10	13	15.5	14.3	64.4	43.3
873	Kubanka	61.4	1 HAD	5	—	5	15.2	14.1	65.0	45.2
882	Ld 134	62.1	2 HAD	3	3	6	14.9	13.5	64.8	44.7
875	Ld 34	62.2	3 HAD	3	5	8	14.5	12.9	65.3	43.1
877	Ld 102	61.9	4 HAD	2	10	12	14.5	12.9	63.3	43.9
881	Ld 133	62.7	3 HAD	5	5	10	14.3	13.1	64.8	44.5
879	Ld 105	62.9	3 HAD	4	6	10	14.2	12.8	66.3	46.0
874	Monad	61.4	1 HAD	6	2	8	14.1	12.6	64.3	43.8
883	RL 1317	62.3	2 HAD	5	3	8	14.1	12.8	66.8	45.0
878	Ld 104	62.7	3 HAD	6	7	13	14.1	12.8	64.1	43.4
872	Mindum	62.0	3 HAD	12	7	19	14.0	12.6	65.2	44.9
880	Ld 111	62.1	5 HAD	5	12	17	14.0	12.4	65.3	46.1

[1] Data on 13.5% moisture basis.
[2] HAD = Hard Amber Durum, RD = Red Durum.

in the durum area of the state if found superior in agronomic and macaroni-quality characteristics to the durums that are now being grown. These varieties are denoted by numbers as they have not yet been named. Mindum and Kubanka have proved to be fairly satisfactory in agronomic and quality factors and are the varieties which are at present in general production. The test weight per bushel varies from 64.6 to 60.8 lbs. The grade varies from No. 1 Hard Amber Durum to No. 5 Hard Amber Durum, the lower grades being found without exception in the Langdon series.

The effect of fungus infections in 1940 is evident in the damaged-kernel percentages. The light kernel damage classification contained the kernels which showed tip discoloration without visible damage in the crease or other parts of the kernel. The evidence of this form of damage can be almost entirely removed by rubbing the infected part of the kernel. The heavy-damage classification comprised kernels with more of the surface, including the crease, showing injury. Total kernel damage is the sum of light and heavy kernel damage. Some variability in wheat protein among the samples is evident, especially in the 1939 Fargo series. These differences must be ascribed to differential varietal responses to environmental conditions of soil and climate. The semolina protein is less variable. The yields of unpurified and purified semolina are both given in the table, the latter values of course being much lower than the former as a result of removal of bran and fibrous material during purification. The number of specks was greatly reduced by purification, while the color was correspondingly improved.

Table II presents the absorption and quality ratings of the semolina and macaroni. The number of specks in the semolina is greatly increased in the 1940 Langdon samples, but on the other hand a noticeable amount of damage on the 1940 Fargo wheats was not reflected in increased semolina speckiness. This was no doubt owing to the fact that the damage had not penetrated through the bran to damage markedly the milling quality of the kernel. The absorptions are fairly consistent and do not appear to have been greatly affected by the unfavorable weather conditions at Langdon. A marked range in color score of macaroni is noticeable, varying from 2.0 to 9.0 at Fargo, 1939; from 2.0 to 8.0 for Fargo, 1940; and from 2.0 to 5.0 for Langdon, 1940. In addition, the Langdon samples were more or less brown and pale in color and would be unsatisfactory for commercial grade long goods.

The varietal color scores are presented in Figures 7 to 9. In each figure the data obtained from one series is represented. Figure 7 presents the Fargo 1939 color scores, while Figure 8 presents similar results for 1940. The Langdon results for 1940 are presented in Figure 9. It will be noticed that Mindum was first in the group in color rating

TABLE II

ABSORPTION AND QUALITY RATINGS OF THE SEMOLINA AND MACARONI

(Results arranged in order of increasing macaroni color score within stations)

Lab. No.	Variety	Semolina			Visual color score of macaroni, perfect score 10 [1]
		Specks per 10 sq. in.	Rating within stations	Absorption	
		no.		%	
		FARGO, 1939			
39–642	Monad	50	7	27.2	2.0 Pk
643	Golden Ball	32	4	28.1	2.0 Pk
644	Pentad	78	8	28.0	2.0 Pk
641	Kubanka	44	6	27.4	5.0
645	Kubanka 49	30	3	28.2	7.0
646	Kubanka 314	20	1	28.1	7.0
648	Ld 34	28	2	28.1	8.0
647	Kubanka 74	32	4	28.0	9.0
649	Ld 9 × Min	34	5	28.1	9.0
640	Mindum	30	3	27.2	9.0
		FARGO, 1940			
40–895	Pentad	42	9	27.2	2.0 Br
901	Kubanka 49	26	7	27.4	5.0
902	Kubanka 314	22	6	27.4	5.0
894	Kubanka	12	2	27.7	6.0
896	Ld 34	10	1	27.5	6.0
897	Ld 102	18	4	27.2	6.0
900	Ld 134	30	8	27.7	6.0
893	Mindum	22	6	27.4	8.0
898	Ld 104	20	5	27.6	8.0
899	Ld 111	16	3	27.8	8.0
		LANGDON, 1940			
40–874	Monad	32	1	28.8	2.0 Br
875	Ld 34	54	3	28.0	4.0 Br
877	Ld 102	102	8	28.0	4.0 Br
881	Ld 1333	64	4	27.8	4.0 Br
882	Ld 134	102	8	28.1	4.0 Br
883	RL 1317	64	4	27.8	4.0 Br
872	Mindum	94	7	27.3	4.0 P
876	Ld 101	82	6	28.1	4.0 P
879	Ld 105	70	5	28.0	4.0 P
880	Ld 111	126	10	28.0	4.0 P
878	Ld 104	112	9	28.1	4.5 Br
873	Kubanka	42	2	28.7	5.0

[1] Br = brown; Pk = pink; P = pale.

at Fargo in both years. At the Langdon station, on the other hand, Kubanka was first in 1940. Ld 104 was also high, being equal to Mindum at Fargo and second to Kubanka at Langdon, while Ld 111 was in the first classification at Fargo and third in Langdon. These

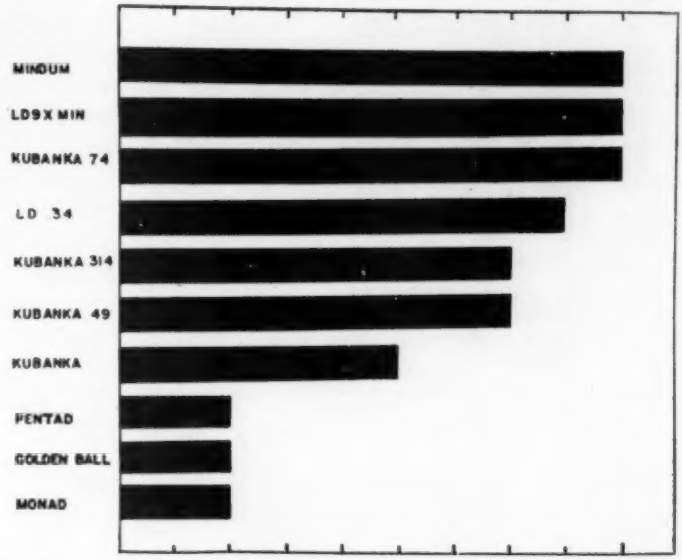


Fig. 7. Visual color score of macaroni processed from durum wheat grown at Fargo in 1939.

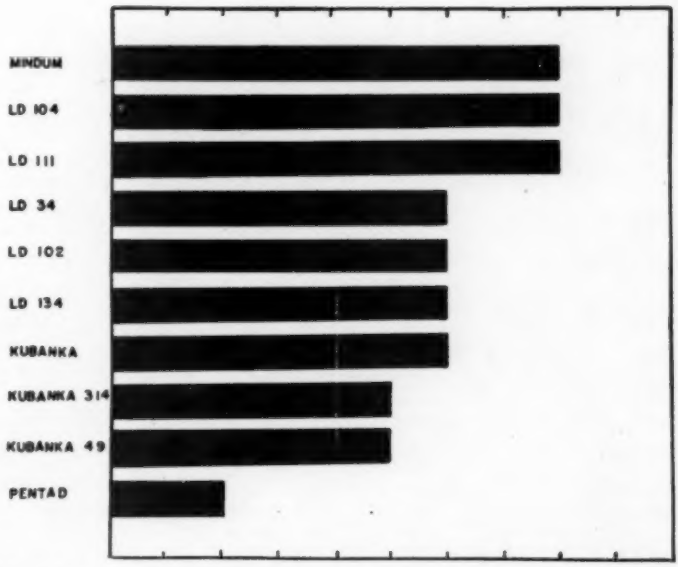


Fig. 8. Visual color score of macaroni processed from durum wheat grown at Fargo in 1940.

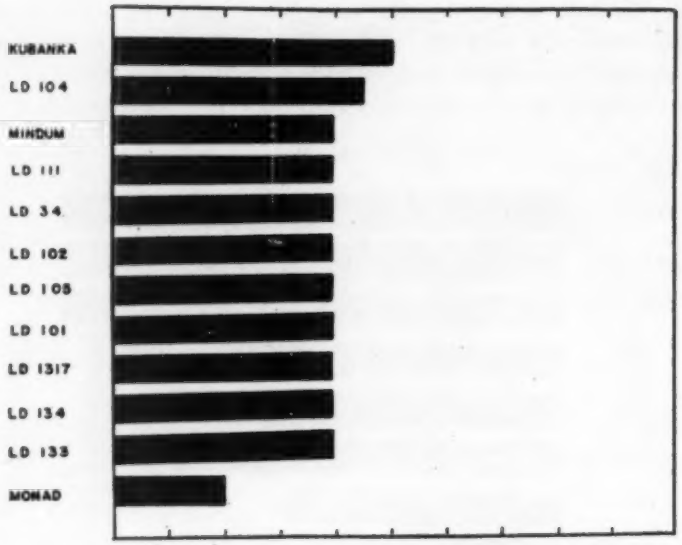


Fig. 9. Visual color score of macaroni processed from durum wheat grown at Langdon in 1940.

two varieties were not included in the 1939 Fargo samples. Ld 34, which was second highest in Fargo in 1938 (Harris and Knowles, 1940), was second in Fargo in 1939 and also in the second group in 1940. Monad, Golden Ball, and Pentad were at the foot of the list wherever grown in 1938, 1939, and 1940. This result is in agreement with their accepted quality rating. No doubt the results obtained from the 1940 samples grown at Langdon have been vitiated as a result of wheat damage. It is also probable that the Fargo values were also adversely affected to some extent, although the visual damage was very much smaller.

In Table III are shown the correlation coefficients calculated between several pertinent variables. Wheat protein and semolina pro-

TABLE III

CORRELATION COEFFICIENTS COMPUTED FROM THE DATA

(Significant coefficients in bold type)

Variables correlated		Correlation coefficients	Probability *P*
x	*y*		
Wheat protein, %	Semolina protein, %	**+.8797**	**<.0001**
Wheat protein, %	Semolina absorption, %	**+.5317**	**.0060**
Test weight, *lbs per bu*	Semolina yield (purified), %	+.0021	>.5485
Semolina yield (unpurified), %	Semolina yield (purified), %	**+.4510**	**.0178**
Semolina specks	Visual macaroni score	**−.4910**	**.0105**
Test weight, *lbs per bu*	Semolina yield (unpurified), %	**+.3935**	**.0365**

tein are very significantly correlated as would be expected by anyone familiar with durum wheat technology. Wheat protein and semolina absorption are also significantly and positively correlated. This is an interesting relationship, as it shows that semolina milled from relatively high-protein durum wheat will take more water to produce a dough of standard consistency. Test weight per bushel was not related to purified semolina yield in the results obtained in this study but was significantly related to yield of unpurified semolina. It is probable that the relatively large number of durum varieties included in the study materially decreased the correlation between test weight and semolina yield. The yield of purified semolina was positively correlated with the yield of unpurified semolina. This relationship, however, was not of sufficient magnitude to permit the prediction of one variable from the knowledge of another. The relationship between the semolina speckiness and visual macaroni score was also determined, although these were subjective measurements. A significant negative correlation was found between these variables, but the magnitude of this correlation was not sufficiently great to be of marked utility in predicting one variable from a knowledge of the other.

Summary and Conclusions

Milling and processing equipment for durum wheat at the North Dakota Experiment Station has been described in some detail, and the techniques employed in the quality evaluation of 32 samples of durum wheat outlined.

The equipment consists of a two-stand Allis-Chalmers experimental mill fitted with suitable rolls, a macaroni processing unit comprising a mixer, kneader, and press, the latter fitted with a device for accurately controlling the press temperature, and a drying cabinet equipped with accessories which enable a time-humidity gradient to be established during the drying period.

A description of the flow sheet used in durum milling is included, as well as a time-humidity gradient chart showing the various relative humidities obtained during the drying period.

Thirty-two samples of durum wheat grown at Langdon and Fargo in 1939 and 1940 were milled and processed by the equipment and methods described in this paper. These samples included varieties which have been shown to have satisfactory quality performance as well as new varieties now under examination with the purpose of possible release later for general production, provided the agronomic and macaroni-making qualities are satisfactory. A few undesirable varieties were also examined to obtain data in comparison with the other wheat studied.

The results of the 1940 wheats grown at Langdon were markedly affected by damage caused by unfavorable weather conditions preceding and during harvest. Injury from various forms of blight, bacterial infections, weathering, etc. was reflected in kernel discoloration, semolina speckiness, and visual color score of macaroni.

Mindum was in the first group for macaroni color in Fargo for both seasons, while Kubanka was among the first in 1940. Ld 104 was another variety which had relatively high macaroni color scores at both stations while the second new wheat, Ld 111, was next. Both wheats showed excellent promise.

Wheat and semolina protein were highly correlated, with wheat protein being related to a lesser degree with semolina absorption. Test weight per bushel was positively related to yield of unpurified semolina but not with yield of purified semolina. Semolina speckiness was inversely related to macaroni color scores.

Literature Cited

Binnington, D. S., and Geddes, W. F.
1936 Experimental durum milling and macaroni-making technique. Cereal Chem. **13**: 497–521.
1937 The relative macaroni-making quality of a number of durum wheat varieties. Cereal Chem. **14**: 293–304.
1939 Further studies upon the relative macaroni-making quality of a number of durum wheat varieties. Cereal Chem. **16**: 384–392.
Fifield, C. C.
1934 Experimental equipment for the manufacture of alimentary pastes. Cereal Chem. **11**: 330–334.
—— Smith, G. S., and Hayes, J. F.
1937 Quality in durum wheats and a method for testing small samples. Cereal Chem. **14**: 661–673.
Harris, R. H., and Knowles, Darline
1940 Quality studies on North Dakota durum wheats (1938 crop). Cereal Chem. **17**: 480–490.
—— and Sibbitt, L. D.
1941 Blight and other damage of wheat in 1940. (I) Effect on macaroni-making quality. N. D. Agr. Exp. Sta. Bul. 296.

THE QUALITY OF NORTH DAKOTA DURUM WHEAT AS AFFECTED BY BLIGHT AND OTHER FORMS OF DAMAGE IN 1940[1]

R. H. HARRIS and L. D. SIBBITT[2]

North Dakota Agricultural Experiment Station, Fargo, North Dakota

(Received for publication September 26, 1941)

During the ripening and harvest season of 1940 the weather conditions in the North Dakota durum-growing region were very favorable for the growth of various microörganisms upon durum wheat, while incipient sprouting, weathering, and similar factors also injuriously affected durum quality. These various forms of damage combined to decrease the grade of the wheat and, in many instances, prevented Federal loans from being made because the grade fell below the minimum allowable for loan purposes. Little information is available in the literature regarding the effects of damage by these causes upon the quality of macaroni.

One of the chief sources of worry to the durum trade in respect to the effects of unfavorable weather upon durum quality is the discoloration of more or less of the surface of the kernel caused by the fungus *Helmenthosporium*. This condition is known as "black point" in common parlance. The development of this fungus is favored by the accumulation and retention of moisture at the germ end of the kernel. Buyers of durum wheat have been convinced for some time that "black point" infection in durum wheat has caused substantial damage to quality. The tendency is for the commercial grain buyer to avoid any section in which wheat damage has been reported. The difficulty occasioned by the infection lies in effecting a clean separation in milling; the semolina contains black specks which in turn exert a degrading influence upon the macaroni. Some chemists have thought that a small degree of contamination is permissible, without injury to semolina color, but extreme care would have to be taken regarding the quantity allowed in the mix as well as in the degree of damage of individual kernels. Available information has indicated that the presence of very small proportions of completely discolored kernels is much more serious than partly damaged kernels. There is also evidence that black-tipped and black-creased kernels are about equally effective in regard to semolina color.

Brentzel (1941) found two types of damage present in the 1940 North Dakota durum wheat crop. These were respectively classified

[1] Published with the approval of the Director of the North Dakota Agricultural Experiment Station.

[2] No seniority in authorship is implied.

as "black point" and "other types." Black point was discovered in many samples, and consisted of two forms of similar appearance which could not be separated by visual inspection. Cultural and microscopic examinations, however, showed that most of the black point was caused by species of the fungus *Alternaria*, while a smaller portion resulted from the presence of *Helmenthosporium sativum*. Kernels affected by the former were often plump and heavier than unaffected seeds. There was no noticeable shriveling caused by this fungus, but germination was somewhat impaired. Kernels attacked by *Helmenthosporium*, on the other hand, were somewhat shriveled.

The "other types" comprised all defects except black point. A little scab (*Gibberella*) was present with some bacterial infection and a number of molds. Damage due to weathering, sprouting, shriveling, etc., was also present.

To evaluate the effects of this damage upon macaroni-making quality Harris and Sibbitt (1941) conducted a preliminary investigation upon a small number of samples which were available, using a modified form of the standardized procedures described by Binnington and Geddes (1936) with the exception that a scientifically controlled drying cabinet was not used for drying the macaroni. A second small series of samples was prepared from blends of light and heavily damaged wheat mixed with the same wheat with the damage removed. Because of the time required to separate the wheat into the various portions it was not possible to procure sufficient grain for the standard durum milling and processing technique, and accordingly the micro method described by Fifield, Smith, and Hays (1937) was employed. This method requires only 100 g of wheat, and consists essentially in milling a relatively small quantity of wheat according to the methods used in milling the customary-sized sample with slight modifications, mixing to a stiff dough, kneading by repeatedly passing the dough through a pair of manually operated steel rolls, pressing, and drying. A hydraulic press is used for the pressing and the disks are then dried. This method gives results which compare favorably, in respect to color, with values yielded by the standard technique.

The results obtained from the investigation showed that the chief effect of the damage was to increase the number of specks in the semolina and to decrease the semolina and macaroni color. The yield of semolina was also decreased. Heavily damaged kernels which showed extensive surface and crease injury had the greatest effect in decreasing quality, but light injury, visible only at the tip and removable by rubbing, if present in sufficient quantity, increased the speckiness and decreased the color ratings.

The conclusions derived from this tentative study convinced the authors that a further investigation should be made, using a larger number of samples which would cover a wider range of injury. It was also felt that additional information could be obtained by employing the standardized technique described by Harris and Sibbitt (1942) since an experimental drier with controlled temperature and humidity was now available. Suitable assistance was secured from the WPA to separate the wheat into the different classifications desired in the investigation and the following study undertaken.

Experimental Material and Methods

A large sample of damaged durum wheat was obtained from the territory in which wheat injury was prevalent. This sample graded (unofficially) No. 5 Hard Amber Durum and contained 28% damaged kernels. As in the previous investigation the total kernel injury was divided into light and heavy damage for the purposes of the investigation. All injured kernels were calculated as per cent by weight of the total. The classification of damage was done by two operators who worked under the same source and intensity of illumination throughout the project. Before making up the blends, the various separations were thoroughly reëxamined by experienced operators, and any kernels that did not appear to be properly placed were reclassified. It is felt that the separations were as truly representative of the indicated degree of injury as it was possible to obtain. The various proportions of damaged wheat included in the blends were chosen to yield as much information as possible upon the effect of the amount of damage upon the quality of the wheat, semolina, and macaroni. The percentages of infected wheat were accordingly varied from 5% to 75% by weight. These blends were made with a good-quality durum grown in 1939. The various samples were thoroughly mixed before sampling and milling.

The different lots of blended wheat were analyzed for moisture and protein content. Test weight per bushel and grade were determined and 3,000 g of the wheat were taken for milling into semolina. The milling was done on a double-stand Allis-Chalmers experimental mill fitted with Allis-Chalmers 19th middlings cut rolls running dull to dull with ¾-inch spiral. One pair of rolls contained 16 while the other had 24 corrugations per linear inch.

The flow sheet used corresponds to the one illustrated by Harris and Sibbitt (1942).

Discussion

A description of the blends used in this investigation with associated data are shown in Table I. The samples are arranged in order of increasing macaroni color score. The test weight and grade tend to decrease with an increase in percentage of damaged wheat included in the blend, particularly if the damage was heavy. The test weight fell from 62.2 to 58.8 lbs per bushel when 50% of heavily damaged kernels was included in the mix, while the grade decreased from No. 1 Hard Amber Durum to Sample Grade Durum. The protein of wheat and semolina tends to increase with addition of damaged wheat, regardless

TABLE I

WHEAT DESCRIPTION, PROTEIN, AND MILLING DATA ON DAMAGED DURUM WHEATS

(Results arranged in order of increasing macaroni color score)

Lab. No.	Description of blend [1]	Test wt. per bu.	Unofficial grade	Protein (N × 5.7) [2]		Semolina yield [2]	
				Wheat	Semolina	Unpurified	Purified
		lbs		%	%	%	%
40–908	50% heavy	58.8	SGD	14.6	12.9	64.3	45.5
909	25% heavy	60.4	SGHAD	13.8	12.6	64.6	45.4
910	10% heavy	61.7	4 HAD	13.5	12.2	65.2	45.7
906	Original (16% light, 12% heavy)	61.1	5 HAD	14.9	13.0	65.9	45.3
912	75% light	61.5	1 HAD	14.2	12.8	64.9	46.0
911	5% heavy	61.9	3 HAD	13.4	12.1	64.9	44.9
913	50% light	61.7	1 HAD	13.9	12.6	66.0	45.2
914	25% light	61.8	1 HAD	13.6	12.4	64.1	44.5
915	10% light	62.0	1 HAD	13.2	12.1	63.3	43.6
916	5% light	62.1	1 HAD	13.2	12.0	64.0	44.6
870	No visible damage	62.9	1 HAD	13.1	12.3	65.9	46.8

[1] Blends made with 40–870 plus indicated percentage of damaged wheat.
[2] Data on 13.5% moisture basis.

of whether the damage was light or heavy. This was caused by the higher percentage of protein in the damaged wheat. No consistent trend in semolina yield was evident when the proportion of damaged wheat in the blend was changed.

In Table II are shown the speck count of the semolina, semolina quality rating, absorption, and visual color score. The increase in number of specks per ten square inches with increase in amount of damage is very clearly shown. There was little difference in absorption among the semolinas manufactured from the various blends, and apparently this property was not affected by wheat damage of this nature. The effect of percentage of damaged kernels upon macaroni color is very evident in the data, especially in the case of heavy kernel damage.

TABLE II

ABSORPTION AND QUALITY RATINGS OF THE SEMOLINA AND MACARONI

Lab. No.	Description of blend [1]	Semolina [2]			Visual color score of macaroni [2] (perfect score 10)
		Specks per 10 sq. in.	Rating	Absorption	
				%	
40–908	50% heavy	220	10	27.6	1.0 br mottled and specky
909	25% heavy	134	9	28.1	3.0 br mottled and specky
910	10% heavy	106	7	27.6	4.0 br mottled and specky
906	Original (16% light, 12% heavy)	130	8	28.1	4.5 br mottled
912	75% light	64	6	28.1	5.0 sl br mottled
911	5% heavy	50	4	28.0	8.0 mottled
913	50% light	60	5	28.0	8.0 mottled
914	25% light	40	3	28.2	8.0 mottled
915	10% light	16	1	28.3	9.0 sl mottled
916	5% light	16	1	28.2	9.0
870	No visible damage	28	2	27.7	9.0

[1] Blends made with 40–870 plus indicated percentage of damaged wheat.
[2] Data on 13.5% moisture basis.
[3] Br = brown; Sl = slightly.

In Figure 1 the effects of the different percentages of light and heavy damage upon test weight, semolina speckiness, and macaroni color score are shown graphically. The influence of the amount of heavy damage upon these three factors is strikingly brought out and illustrates the care required to control rigidly the proportion of heavily damaged kernels allowed in a durum mix. Five per cent of such damage seriously lowered the color of the macaroni, while 10% was extremely detrimental to macaroni color as well as to semolina speckiness. It also adversely affected the test weight per bushel. The inclusion of 50% of heavily damaged kernels decreased the test weight to less than 59 lbs, increased the number of specks per ten square inches to well over 200, and reduced the macaroni color score to a very low value. While no commercial mill would consider using a mix containing such a high proportion of this form of damage, the data are useful in showing the extremely adverse effects of the blend.

The light kernel damage had much less effect upon the three factors represented. Test weight, while slightly reduced, did not fall below 61 lbs even when 75% of light damage was present. The number of semolina specks also was increased only very slightly. The greatest effect of this form of damage was found to be upon the macaroni color, but when the percentage of damaged kernels was below 10% there was apparently no effect and 25% did not seriously degrade the color. When 50% was present, however, the color commenced to fall off

rapidly, and when 75% was reached the macaroni was decidedly inferior.

Figure 2 represents the effect of the various percentages of damaged

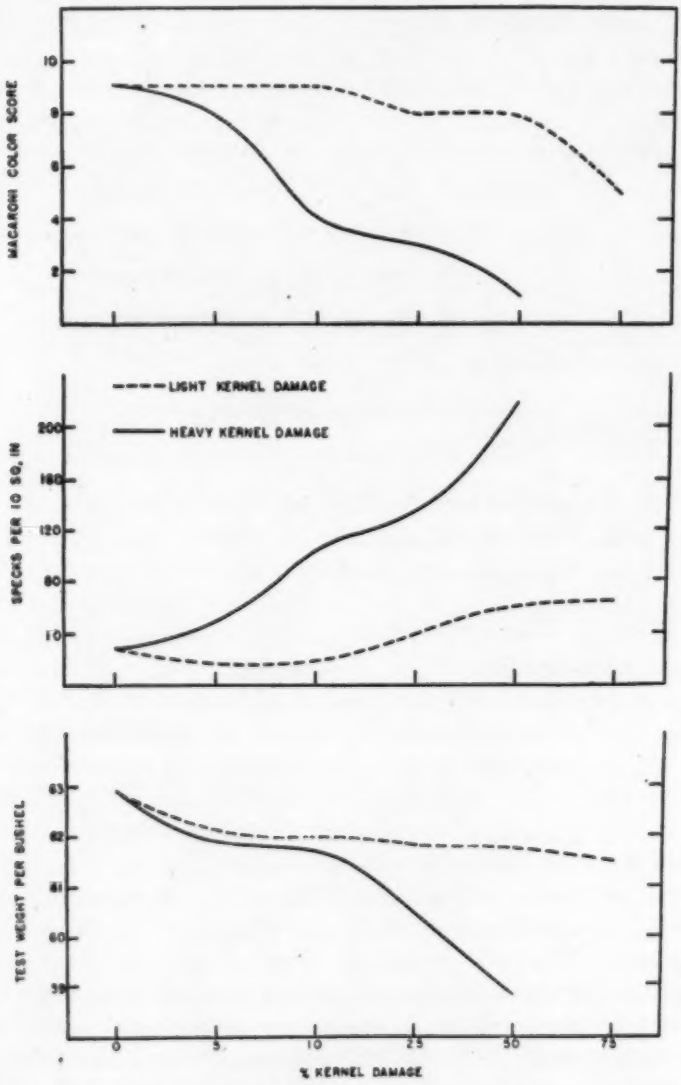


Fig. 1. Effect of heavy and light kernel damage upon test weight, semolina specks and macaroni color.

kernels upon macaroni color. The data are arranged in order of decreasing color score from left to right, and emphasize the conclusions already reached in respect to the influence of heavy kernel damage

upon this macaroni property. No degrading effect was noticeable with light damage until 25% concentration was reached, but as no blends were made between 10% and 25% some slight influence upon color may have been exerted at a lower proportion of damaged kernels. Twenty-five and 50% light, as well as 5% heavy damage, gave the same macaroni color score. Seventy-five per cent light damage markedly reduced the color while more than 5% of the heavy damage was extremely injurious.

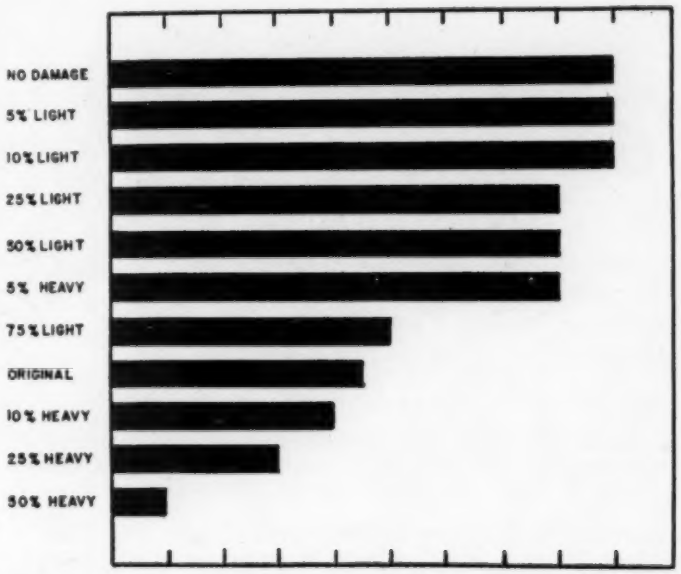


Fig. 2. Effects of different percentages of light and heavy kernel damage upon macaroni color score.

Summary and Conclusions

The belief of many chemists regarding the detrimental effects of different degrees of damaged durum wheat in the mill mix upon semolina and macaroni quality has been largely substantiated by the present investigation. Lightly damaged kernels showing discoloration at the tip without apparent injury to the other portions of the kernel can be tolerated in as high a proportion as 10% with good milling durum, while 25% does not greatly lower macaroni color or increase semolina speckiness. Higher proportions than 50% would be extremely hazardous to use in the mill mix.

The situation when heavily damaged kernels showing evidences of injury in the crease and other portions of the kernel are included in the blend is more critical. The presence of 5% of damaged grain signifi-

cantly affects the number of semolina specks and macaroni color score, while 10% is very detrimental.

In milling durum wheat damaged by "black point" and other infections special care is required in respect to the degree of damage of individual kernels permitted in the mill blend. If only light injury at the tip of the kernel is present the situation is not critical so far as proportion of damaged wheat is concerned, but a careful check should be kept upon the quality of the products while the blend is being milled. If heavy kernel damage is present, extreme diligence should be exercised to keep the quantity allowed to go to the mill at any time below 5%. It must also be remembered that if light as well as heavy damage is present, the effect upon quality will be additive and greater attention will have to be paid to the maximum quantity of heavily infected kernels allowed.

It was found that the grade was materially lowered by the presence of heavily damaged kernels, the addition of 50% resulting in decreasing the grade from No. 1 Hard Amber Durum to Sample Grade Durum. This would entail a serious financial loss to the grower. The presence of 5% of heavily injured grain lowered the grade to No. 3 Hard Amber Durum. Light damage was without appreciable effect upon the grade under the existing Federal grading regulations.

Acknowledgment

The authors wish to acknowledge assistance provided by the Work Projects Administration, Research and Records Division, through the operation of Seed Testing Project O.P. 165–1–73–144, during this investigation.

Literature Cited

Binnington, D. S., and Geddes, W. F.
1936 Experimental durum milling and macaroni-making technique. Cereal Chem. **13**: 497–521.

Brentzel, W. E.
1941 Blight and other damage of durum wheat in 1940. (2) Effect on grain to be used for seed. N. D. Agr. Exp. Sta. Bul. 296.

Fifield, C. C., Smith, G. S., and Hayes, J. F.
1937 Quality in durum wheats and a method for testing small samples. Cereal Chem. **14**: 661–673.

Harris, R. H., and Sibbitt, L. D.
1941 Blight and other damage of durum wheat in 1940. (1) Effect on macaroni-making quality. N. D. Agr. Exp. Sta. Bul. 296.
1942 Experimental durum milling and processing equipment with further quality studies on North Dakota durum wheats. Cereal Chem. **19**: (previous paper, this issue).

THE INFLUENCE OF THE DRYING PROCEDURE ON MALT COMPOSITION [1]

ALLAN D. DICKSON and H. L. SHANDS [2]

Division of Cereal Crops and Diseases, Bureau of Plant Industry, U. S. Department of Agriculture, and University of Wisconsin, Madison, Wisconsin

(Received for publication November 3, 1941)

The influence of drying procedure on certain characteristics of finished malt has been known and used commercially for many years. Previous to 1900 Prior, as reported in Wahl and Henius's *Handy Book* (1902), studied this subject rather extensively with particular emphasis on the effect of drying temperature on some enzyme systems.

More recent literature agrees that drying reduces enzymatic activities, particularly diastatic power, but is contradictory on the effect of drying on color formation and the solubility of various nitrogen and carbohydrate constituents. The contradictions are not surprising since Kolbach and Schild (1935) showed that the effect of higher temperatures was largely dependent on the moisture content of the malt at the time high temperatures were applied.

In investigating the malting quality of different barley varieties, it was found that the drying procedure appreciably affected malt composition and was of importance in the evaluation of barleys. From 1935 to 1937, limited studies of drying procedures have been made at the Malt Laboratory at Madison. The results suggested an experiment where green malts produced under identical conditions were given drying treatments so planned that small changes in composition might be more easily interpreted.

Materials and Methods

The first study was made in 1939 on Wisconsin Barbless and Oderbrucker barleys grown in that year. Both were field samples, the Wisconsin Barbless from University Farm and the Oderbrucker from a farm near Madison. Quantities of the barleys were subdivided into 170-g samples (on a dry basis), and these were steeped to and malted at approximately 46% moisture for six days at 16°C (60.8°F). The malting was done in the small experimental chamber described by Shands, Dickson, and Dickson (1941). At the end of six days, one sample was used for green malt analysis, and the others were placed in

[1] Based on cooperative investigations between the Division of Cereal Crops and Diseases, Bureau of Plant Industry, U. S. Department of Agriculture, and the Wisconsin Agricultural Experiment Station. The United States Maltsters Association has cooperated through an industrial fellowship grant to the University of Wisconsin.

[2] Agent, Division of Cereal Crops and Diseases, Bureau of Plant Industry, U. S. Department of Agriculture, and Assistant Professor of Agronomy, Wisconsin Agricultural Experiment Station.

the dryer. After they had received certain specified drying treatments, samples were removed, cleaned, and placed in friction top cans until analysis could be made. The drying treatment for each malt is shown in Table I. These drying conditions were designed to reduce moisture to a reasonably low level before the temperatures were increased. Samples of the malts were removed from the dryer at short intervals in order to follow changes in composition more closely.

The first series of malts was analyzed for moisture, diastatic power, extract, color, and the various nitrogen fractions of the laboratory worts. The green malts were frozen with dry ice and ground through a chilled food chopper. The ground malt was allowed to thaw and dry slightly before samples for moisture and diastatic power determinations were taken.

The methods of the American Society of Brewing Chemists (1937) were used, except for diastatic power, where the ferricyanide modification was used. The nitrogen fractions were determined according to the methods described by Dickson and Burkhart (1942).

The study was repeated the following year with two other samples of Oderbrucker and Wisconsin Barbless barley grown in farmers' fields in Wisconsin in 1940. Minor modifications in the drying temperatures and times were used as indicated in Table II. After a short preliminary kilning, two samples were dried further by phosphorous pentoxide at room temperature in a vacuum desiccator. These malts were analyzed in more detail, including total diastatic activity with papain extraction, alpha-amylase by the method of Blom and Bak (1938), and proteolytic power by a modification of the method of Kolbach and Simon (1936).

Drying Treatments in Kiln

The data on the first series, carried out in 1939, are given in Table I. In Table II are given the more detailed data obtained on the 1940 series of malts. The relation between moisture loss and the reduction of diastatic power during drying for the two varieties and the two years is shown graphically in Figure 1.

In three out of four malts, the diastatic value on the green malt was appreciably below that of a similar malt which had been dried at a relatively low temperature. The evidence indicated that this was caused by incomplete enzyme extraction from the green malt, rather than a further enzyme production during the early stages of drying. Drying was rapid enough to preclude any great increase in enzyme activation during the process, since the quantities of malt were small and the air velocity was high. Other work on the determination of diastatic power in green malt has indicated the difficulty of grinding

TABLE I

THE INFLUENCE OF TEMPERATURE AND TIME OF DRYING ON THE COMPOSITION OF MALTS MADE FROM ODERBRUCKER AND WISCONSIN BARBLESS BARLEYS—1939 SERIES

Drying treatment [1]	Barley variety	Moisture	Diastatic power	Extract (dry basis)	Color	Wort nitrogen
		%	°L	%	Lov. 52	%
None Green malt	Oderbrucker	47.1	207	—	—	—
2 hrs 45°C (113°F)	"	11.0	226	73.2	1.6	0.77
12 hrs 45°C (113°F)	"	6.1	221	72.9	1.9	0.78
12 hrs 45°C + 4 hrs 55°C (131°F)	"	5.6	218	72.7	1.9	0.76
12 hrs 45°C + 4 hrs 55°C + 4 hrs 65°C (149°F)	"	4.5	193	73.0	1.8	0.77
12 hrs 45°C + 4 hrs 55°C + 6 hrs 65°C	"	4.6	205	72.8	1.9	0.76
12 hrs 45°C + 4 hrs 55°C + 6 hrs 65°C + 2 hrs 75°C (167°F)	"	3.7	165	72.6	1.9	0.77
12 hrs 45°C + 4 hrs 55°C + 6 hrs 65°C + 2 hrs 75°C + 2 hrs 85°C (185°F)	"	3.2	135	72.3	2.0	0.77
2 hrs 45°C + 4 hrs 75°C	"	4.5	156	72.8	1.8	0.77
None Green malt	Oderbrucker	46.1	219	—	—	—
2 hrs 40°C (104°F)	"	10.7	217	72.9	1.5	0.74
26 hrs 40°C	"	6.3	221	73.1	1.6	0.75
50 hrs 40°C	"	6.1	230	73.2	1.6	0.77
50 hrs 40°C + 4 hrs 75°C	"	4.0	166	72.6	1.8	0.76
None Green malt	Wis. Barbless	45.6	118	—	—	—
2 hrs 45°C (113°F)	" "	11.3	119	74.8	1.4	0.58
12 hrs 45°C	" "	7.1	119	74.3	1.4	0.65
12 hrs 45°C + 4 hrs 55°C (131°F)	" "	6.3	119	74.6	1.4	0.61
12 hrs 45°C + 4 hrs 55°C + 4 hrs 65°C (149°F)	" "	4.9	107	74.6	1.4	0.61
12 hrs 45°C + 4 hrs 55°C + 6 hrs 65°C	" "	4.7	97	74.6	1.4	0.60
12 hrs 45°C + 4 hrs 55°C + 6 hrs 65°C + 2 hrs 75°C (167°F)	" "	3.9	85	73.9	1.5	0.61
12 hrs 45°C + 4 hrs 55°C + 6 hrs 65°C + 2 hrs 75°C + 2 hrs 85°C (185°F)	" "	3.1	60	73.5	1.6	0.60
2 hrs 45°C + 4 hrs 75°C	" "	4.3	85	74.0	1.5	0.59
None Green malt	Wis. Barbless	45.4	121	—	—	—
2 hrs 40°C (104°F)	" "	11.0	118	74.7	1.4	0.62
26 hrs 40°C	" "	6.9	121	74.3	1.4	0.59
50 hrs 40°C	" "	6.3	112	74.3	1.4	0.61
50 hrs 40°C + 4 hrs 75°C	" "	3.9	75	73.9	1.4	0.59

[1] All samples except green malts received 8 hrs drying at 25°C (77°F) plus 4 hrs at 35°C (95°F), plus additional treatment indicated.

TABLE II

THE INFLUENCE OF TEMPERATURE AND TIME OF DRYING ON THE COMPOSITION OF MALTS MADE FROM ODERBRUCKER AND WISCONSIN BARBLESS BARLEYS—1940 SERIES

Drying treatment[1]					Barley variety	Moisture	Diastatic power	Diastatic power papain digestion,	Alpha-amylase (Blom)	Proteolytic power (Kolbach) total mg of edestin N hyd.	Extract dry basis	Color	Wort N as % of dry malt	Formol wort N as % of wort nitrogen	Maltose value of wort, % of wort solids
Hrs, 45°C 113°F	Hrs, 55°C 131°F	Hrs, 65°C 149°F	Hrs, 75°C 167°F	Hrs, 85°C 185°F											
						%	°L	°L			%	Lov. 52	%	%	
None	Green malt				Oderbrucker	44.7	180	204	79	78	—	—	—	—	—
2					"	10.5	193	234	75	96	75.6	1.3	0.71	13.9	69.0
12					"	7.1	200	251	80	55	75.2	1.1	0.71	16.9	68.9
12	4				"	6.1	197	238	82	54	75.5	1.3	0.71	18.0	69.1
12	4	4			"	4.9	178	228	79	55	75.3	1.3	0.72	17.5	68.3
12	4	6			"	4.4	169	226	81	52	75.2	1.3	0.71	17.6	69.8
12	4	6	2		"	4.2	162	218	80	55	75.2	1.3	0.71	18.0	69.4
12	4	6	2	2	"	3.3	116	155	81	49	75.1	1.3	0.71	18.0	68.5
12	4	6	2	4	"	2.8	105	148	79	57	75.0	1.5	0.71	18.7	67.7
2 + 43 days over P_2O_5—room temp.					"	2.7	176	192	84	52	—	—	—	—	—
None	Green malt				Wis. Barbless	44.3	131	151	39	40	—	—	—	—	—
2					" "	10.7	146	214	43	36	74.2	1.0	0.55	16.8	69.0
12					" "	7.1	141	210	47	39	74.1	1.0	0.56	17.8	69.0
12	4				" "	6.1	133	215	45	39	73.8	1.0	0.55	18.1	68.8
12	4	4			" "	4.9	119	183	46	35	73.9	1.1	0.57	16.3	68.2
12	4	6			" "	4.6	112	175	49	—	73.5	1.3	0.55	17.3	68.8
12	4	6	2		" "	4.3	106	164	42	28	73.6	1.3	0.55	17.3	68.7
12	4	6	2	2	" "	3.5	77	111	42	26	73.7	1.3	0.56	16.4	67.8
12	4	6	2	4	" "	3.1	67	111	41	33	73.6	1.4	0.55	17.1	67.6
2 + 43 days over P_2O_5—room temp.					" "	2.9	122	142	43	37	—	—	—	—	—

[1] All samples except green malts received 8 hrs drying at 25°C (77°F) plus 4 hrs at 35°C (95°F), plus additional treatment indicated.

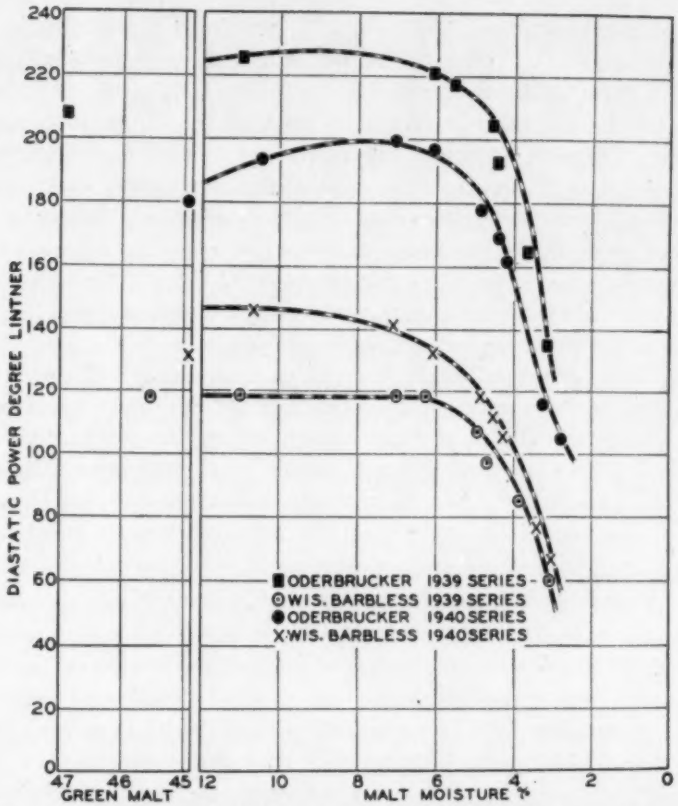


Fig. 1. The relation between moisture loss and the reduction of diastatic power during drying of malts made from Oderbrucker and Wisconsin Barbless barleys.

the sample sufficiently to obtain a complete extraction of the enzymes. The "total" diastatic power values obtained by using papain extraction are further evidence that the size of the particles in the ground material may have been too large to permit complete diffusion of the extraction medium.

At temperatures of 55°C (131°F) and below, when the moisture content of the malts was not reduced below 6%, there was no great reduction in diastatic power. With increasing high temperature and particularly below 5% moisture in the malt, the decrease in diastatic power was very rapid. There appeared to be a critical point in diastatic power when drying conditions reduced the moisture below 6%. In other studies of malting quality of different barley varieties made at the Madison Laboratory, malts have been dried to final moistures of between 5% and 6%. As indicated in this study, this procedure gives a more reliable measure of the potential ability of a variety to develop diastatic power, without introducing the variable of excessive kilning.

Long periods of drying at 40°C (104°F) produced practically no reduction in diastase but reduced the moisture only to approximately 6%. There is some evidence in the data of Table I that the enzymes were more stable to heat when the moisture content of the malt was low. Although the total reduction during drying was appreciably greater in the high-diastatic Oderbrucker malts, the percentage reduction was somewhat greater in the Wisconsin Barbless samples. Kopecky (1937) states that the richer the green malt in diastase, the greater is the loss of diastatic activity on the kiln. Apparently this is true for absolute values, but not when the two varieties, Oderbrucker and Wisconsin Barbless, are compared on a percentage basis.

The data given in Table II appear to substantiate the fact that alpha-amylase was much more stable to heat than beta-amylase, since there was practically no reduction in the former throughout the drying process. The values fluctuated within relatively narrow limits for both varieties and the variation was not consistently associated with drying treatment. It would seem that the reduction in total diastatic value caused by kilning treatments would be accounted for primarily by the inactivation of the beta-amylase component.

The data on proteolytic power, using a modification of Kolbach and Simons' (1936) edestin method, are difficult to interpret. Extraction of the enzymes from the green malt of the Oderbrucker samples appeared incomplete. A large reduction in activity was indicated between 2 and 12 hours' drying at 45°C, with little or no further decrease at the higher temperatures. The highest proteolytic power value on Wisconsin Barbless malts was obtained on the green malt, with a small decrease after a temperature of 65°C was reached. These limited data might indicate that the proteolytic enzyme systems of the varieties are different in their reaction to drying conditions. Heintz (1939) applied different kilning treatments to malts which showed a high germinative activity and found that proteolysis, as measured by the formol titration, required a temperature of 80°C for reduction and even then was not greatly altered by conditions. The results reported herein on Wisconsin Barbless coincide with the findings of Heintz. In other studies by Ayre and Anderson (1939) and unpublished data from this laboratory, a significant positive correlation has been obtained between proteolytic power and wort nitrogen. These data show that wort nitrogen was uninfluenced by the drying conditions. The absence of an apparent high correlation between two factors in the data from the Oderbrucker samples may be interpreted as evidence of the unreliability of the proteolytic power values. These discrepancies indicate that further study of the effect of drying conditions upon proteolytic

activity is desirable. More reliable methods for determining proteolytic activity need to be developed.

Extract content of the malts did not seem to be greatly affected until temperatures of 75°C to 85°C (167° to 185°F) were reached, when a small reduction was noted. The magnitude of the reduction varied from 0.5% to 1.3% when moisture was reduced from about 10% to 4% or less. The average loss was 0.8%. Maltose values of the wort showed reductions about twice as large as those for extract. Under the drying conditions used, no great increase in color took place, but there was a slight increase at a temperature of 85°C.

Total wort nitrogen appeared to be uninfluenced by drying conditions. In the Oderbrucker samples, formol nitrogen increased very slightly and fairly regularly with increased drying. This fact is of doubtful significance, since a similar trend was not apparent in the Wisconsin Barbless samples. There was an indication that at the higher drying temperatures slightly more of the wort nitrogen became permanently soluble. Kolbach and Schild (1935) showed that the changes in the nitrogen constituents were greatly dependent upon the moisture content of malt and kilning temperature. They found that at high moisture and high temperatures the increase in permanently soluble and formol nitrogen was appreciable. In the present study, the moisture of the malt was reduced to approximately 10% before high temperatures were applied. The results agree more closely with those of Trkan and Zila (1937), who applied different kilning treatments to malts containing 9.0% moisture and found that neither time of curing nor finishing temperatures altered the proportion of the individual nitrogen fractions.

The results reported herein point to a probable explanation of the low diastatic values obtained by Dickson *et al.* (1935) for the 1934 malts. It would appear that less severe kilning conditions would be more appropriate in comparing the ability of different barley varieties to develop enzymatic systems during malting.

Obviously the above data apply only to the drying conditions outlined. However, it is felt that the information should be applicable to commercial kilning procedures used in this country in the production of pale malts. The effective air velocity in commercial kilns is probably less than that used here with small quantities of malt. In most commercial houses the moisture of the malt is about 10% when dropped to the lower kiln for high-temperature drying. The results reported should apply to changes taking place during the final finishing of this malt and could be used to modify drying procedures in commercial houses.

Treatments Other Than Kilning

During the drying of malt, both temperature and changes associated with the loss of moisture may be active in reducing enzymatic activity. It was desired to dry malts to a moisture content of about 3% without the use of high temperatures. As noted in Table I, after 50 hours' drying at 40°C the moisture was reduced only to approximately 6%. The diastatic power was reduced very little if at all below the value for green malt. In order to reduce the moisture without the use of heat, a sample of malt from each variety, containing approximately 10% moisture, was dried for 43 days in a vacuum desiccator over calcium chloride and phosphorous pentoxide. The results are given in Table II. The moisture was reduced to less than 3%, but the diastatic-power values were reduced only from 193 to 176 for Oderbrucker and from 146 to 122 for Wisconsin Barbless, or 9% and 16%, respectively, for the two varieties. With these two samples it appears that drying over phosphorous pentoxide produces a proportionately greater reduction of the inactive diastase, liberated by papain, than of the free form. Verification of this would require further study. In some earlier studies of drying at room temperature, five high-diastatic malts containing from 6% to 8% moisture were dried 43 days over phosphorous pentoxide. The percentage reduction of diastatic power varied from 8% to 20% and the final moistures were from 2.5% to 3.0%.

Replicated samples of the same malts were submitted to heat treatments for 6 and 12 hours at 80°C (176°F) in closed metal cans in order to limit the loss of moisture as much as possible. The results were very erratic and the variation between replicates large, but the percentage reduction in diastatic power varied from 40% to 85%. If a comparison of these two treatments can be justified, the data indicate that high temperature is much more important in the reduction of diastatic power than the loss of moisture.

Summary

A series of malts produced from Oderbrucker and Wisconsin Barbless barleys under uniform conditions were submitted to a drying schedule involving increasing temperatures from 25° to 85°C. Samples were removed at intervals in order to study the effect of time and temperature of drying on malt composition. Above 55°C and at moisture contents below 6%, the reduction in diastatic power was rapid. Alpha-amylase was reduced only slightly if at all. Extract, color, and maltose values of the wort were influenced slightly at the higher temperatures. The effect of the drying procedure on the nitrogen fractions of the wort was very slight.

Acknowledgments

The authors acknowledge the assistance of B. A. Burkhart, L. A. Hunt, and W. W. Pregler in preparing and analyzing the malts herein reported. Assistance in the preparations of these materials was furnished by the personnel of the Works Project Administration Project No. 65-1-53-2349.

Literature Cited

American Society of Brewing Chemists
1937 Official methods of analysis. Chicago, Ill.
Ayre, C. A., and Anderson, J. A.
1939 Varietal differences in barleys and malts. VI. Autolytic proteolytic activity of malt and its correlations with wort nitrogen and barley nitrogen fractions. Can. J. Research **C17**: 239–246.
Blom, J., and Bak, A.
1938 Über die Bestimmung von Amylasen durch Verflüssigung von Stärkekleister. Hoppe-Seyler Z. physiol. Chemie **256**: 197–207.
Dickson, J. G., Shands, H. L., Dickson, A. D., and Burkhart, B. A.
1935 Barley and malt studies. I. Developing new varieties of barley for malting and their properties. Cereal Chem. **12**: 596–609.
Dickson, A. D., and Burkhart, B. A.
1942 Changes in the barley kernel during malting: chemical comparisons of germ and distal portions. Cereal Chem. **19**: 251–262.
Heintz, L.
1939 Determination of germinative, diastatic, and proteolytic powers as factors for evaluation of malt. Schweitzer-Brau. Rundschau **50**: 95–109. *Cf.* J. Inst. Brewing **45**: 351.
Kolbach, P., and Schild, E.
1935 Influence of moisture and temperature upon the change of substances during kilning of malt. Wochschr. Brau. **52**: 129–134, 137–141, 147–150.
Kolbach, P., and Simon, H.
1936 Über die proteolytische Kraft von Malzanszügen und Würzen. Wochschr. Brau. **53**: 297.
Kopecky, O.
1937 Change in diastatic power of green malts in the course of kilning. Petit J. brasseur **45**: 78–79. *Cf.* J. Inst. Brewing **43**: 119.
Shands, H. L., Dickson, A. D., and Dickson, J. G., and B. A. Burkhart.
1941 The influence of temperature, moisture, and growth time on the malting quality of four barley varieties. Cereal Chemistry **18**: 370–394.
Trkan, M., and Zila, U.
1937 Allgem. Brauer- u. Hopfen-Z. **77**: 266. *Cf.* Int. Brew. Abstracts **5** (1937): 543–580.
Wahl, R., and Henius, Max
1902 Handy Book of brewing and malting (2nd ed.). Chicago, Ill.

A SIMPLE METHOD FOR THE APPROXIMATE ESTIMATION OF PROTEOLYTIC ACTIVITY

QUICK LANDIS

The Fleischmann Laboratories, Standard Brands Incorporated, New York

(Read at the Annual Meeting, May 1941)

In 1894 Mett [1] allowed gastric juice to act on coagulated egg albumin contained in small open-ended glass tubes and observed that the coagulum was digested from the open end. The rate of digestion was thus a rough measure of the concentration of the enzyme in the gastric juice, provided the rate of diffusion did not become a limiting factor.

[1] Mett, Arch. Anat. Physiol., p. 68, Verda (1894).

This simple method has been applied to the determination of proteinases of the papain type, using gelatin as a substrate. After some preliminary experiments the apparatus shown in Figure 3 was developed. A set of tubes are filled by inverting in a small rack and pipetting the substrate solution at 30°C into the open end of each tube. They are then covered with a beaker to minimize evaporation and placed in the ice box overnight.

From 20 to 40 ml of the enzyme infusion is placed in a 50-ml flask, a prepared tube inserted, and the assembly placed in a thermostat at

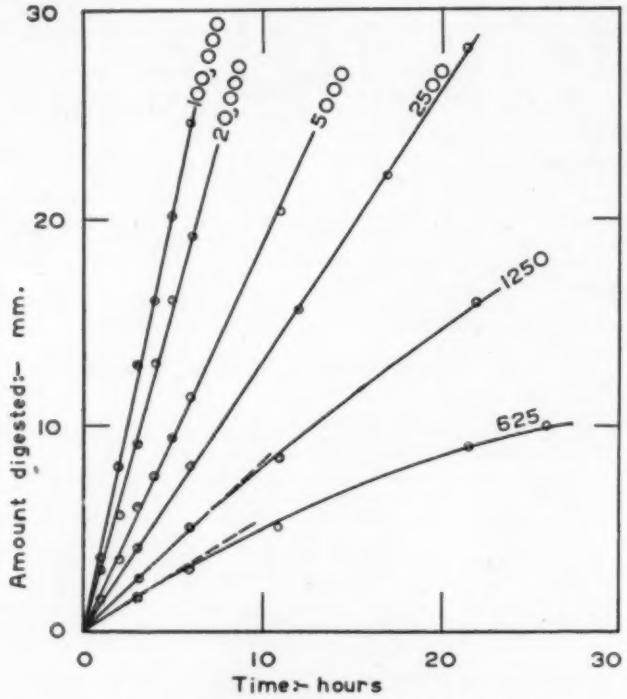


Fig. 1. Rate curves. The concentration of enzyme (ficin) in milliunits per gram (arbitrary scale) is given on each curve.

20°C. After a convenient interval the tube is removed, the digested fluid gently shaken from the end, and the length of material which has been digested is measured. A typical set of curves is given in Figure 1. Under these conditions the amount of digestion is directly proportional to the time over a considerable range.

This method also facilitates the study of activators; since no specified volume of enzyme infusion is required the results are obtained directly in terms of concentration of active enzyme in the infusion. A series of stock solutions can conveniently be used.

The "regular" stock solution which has been found satisfactory is a 0.5*M* solution of NaCl in an acetate buffer solution of 5.0 pH which is 0.5*M* in acetate. Boiled, distilled water is used in making the solutions. Various "activator" stock solutions, with the same buffer and NaCl concentration but 0.05 to 0.3*N* in cyanide, have been used, with suitable precautions to maintain the final NaCl, acetate, and H-ion concentration unchanged. These solutions are diluted 1:10 in preparing either the substrate for the Mett tubes or the enzyme infusions.

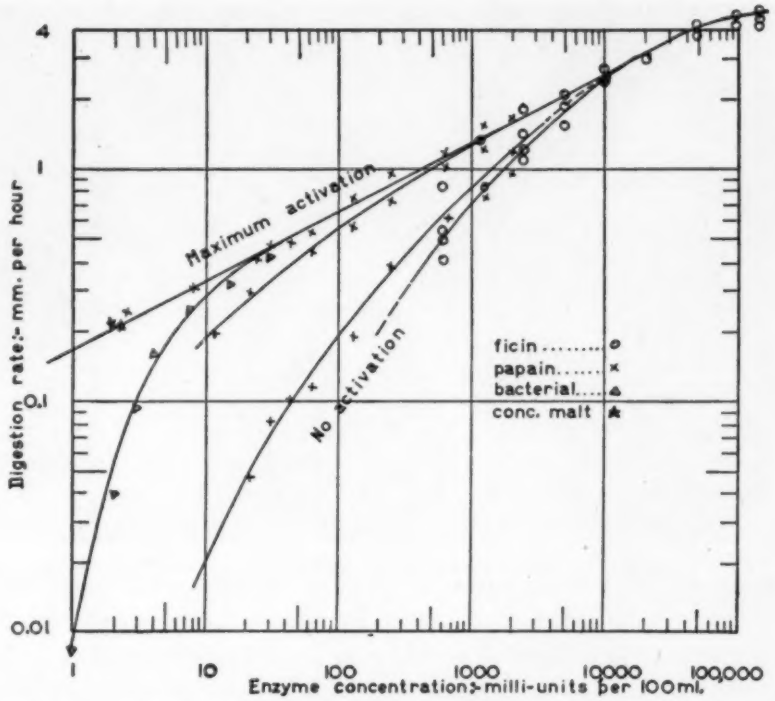


Fig. 2. Concentration relationships. Maximum activation was obtained with the special activator stock solution described in the text.

A concentration of 1.5% gelatin has been found satisfactory for the substrate.

A special activator stock solution was used to obtain the maximum activation as will be shown later. This was prepared from one of the solutions previously described which was 0.3*N* in cyanide. To 500 ml of this solution was added 50 g of ground malt. The mixture was saturated with H_2S and after one hour's gentle maceration the supernatant liquor was centrifuged off and boiled to expel excess H_2S and destroy the malt enzymes. This makes available the natural activators in their reduced form for subsequent experiments.

Some results obtained by this method are indicated in Figure 2. It will be observed that as the enzyme concentrates are diluted their activity diminishes disproportionately. Whether this is due to the presence of inhibitors in the water used for dilution, to an inadequate supply of protective substances in the original material, or to an inherent characteristic of the enzyme, is not clear. However, by the use of maximum activation some enzymes at great dilution can be made to coincide in their activity with that deduced from the more

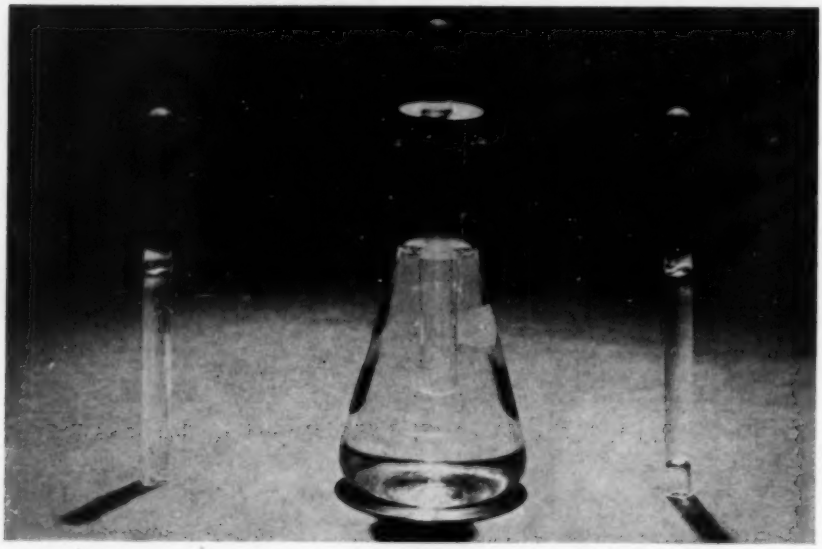


Fig. 3. Apparatus. Glass tubes (6 mm. id) are sealed to glass rods of the same diameter forming a tube closed for half of its length. The gelatin is prevented from melting in the lower end of the tube by the water bath.

concentrated infusions. The common activiators used, however, had no effect on a bacterial protease, although it is possible that its dilution function is different from that of the papain-type enzymes.

Summary

A modified Mett-tube procedure using gelatin for the determination of proteinase concentration is described and an example of its application to the study of activation is given.

HYSTERESIS OF AIR-DRY WHEAT STARCH [1]

HENRY C. REITZ, ROSS AIKEN GORTNER, and RAYMOND E. CARLSON
Division of Agricultural Biochemistry, University of Minnesota, St. Paul, Minnesota

(Received for publication October 14, 1941)

It is well recognized that colloid gels undergo hysteresis or aging with time and that starch pastes show such hysteresis effects to a marked degree. However, we were surprised to find an apparent hysteresis effect taking place in air-dry starch (moisture 10.57%), especially in view of the recommendation of Ripperton (1931) that the viscosity values of different starches should be expressed in terms of a standard starch instead of in absolute viscosity units. Ripperton (1931, p. 154) states that "air-dry starch is very stable in its properties and makes a dependable standard over a period of years." It is the purpose of this note to call this statement in question.

Experimental

A large sample of wheat starch was isolated from *Triticum vulgare* var. Thatcher by centrifuging techniques, the technique being such as to remove all small or broken granules and to yield a pure white air-dry starch containing remarkably uniform granule sizes. The starch as bottled contained 10.57% moisture, 0.041% nitrogen, and 0.180% ash. The ash was almost wholly derived from the phosphoric acid content (0.1588% P_2O_5 in the dry starch). During our experiments the starch has been kept in a sealed glass container.

Viscosity studies were made on the sample by the cold gelatinization technique of Ostwald and Frankel (1927), using NaOH as the gelatinizing reagent. The gelatinization and viscosity-determination procedures were rigidly standardized and all experimental details exactly followed during each series of runs. For the purpose of this note it is not essential to describe these procedures in detail.[2] Suffice it to say that the starches were gelatinized by the appropriate concentration of NaOH at 30°C for a two-hour period. The flasks containing the starch and NaOH solution were slowly turned in a mechanical rotator immersed in the constant-temperature bath during the gelatinization period. At the end of the period the viscosity was determined at 30° ± 0.03°C, using an Ostwald viscometer made of pyrex glass, having a volume of 19.54 ml, a capillary length of 9.76 cm, and a capillary diameter of 0.188 cm. The viscometer employed was carefully standardized against glycerol solutions of known viscosity. The viscosities were calculated in centistokes.

[1] Paper No. 1931, Scientific Journal Series, Minnesota Agricultural Experiment Station.
[2] The techniques are detailed in the manuscript copy of a Ph.D. thesis by H. C. Reitz entitled "A Comparative Study of the Starches of the Triticum Species," University of Minnesota, June, 1938. On file in the Library of the University of Minnesota.

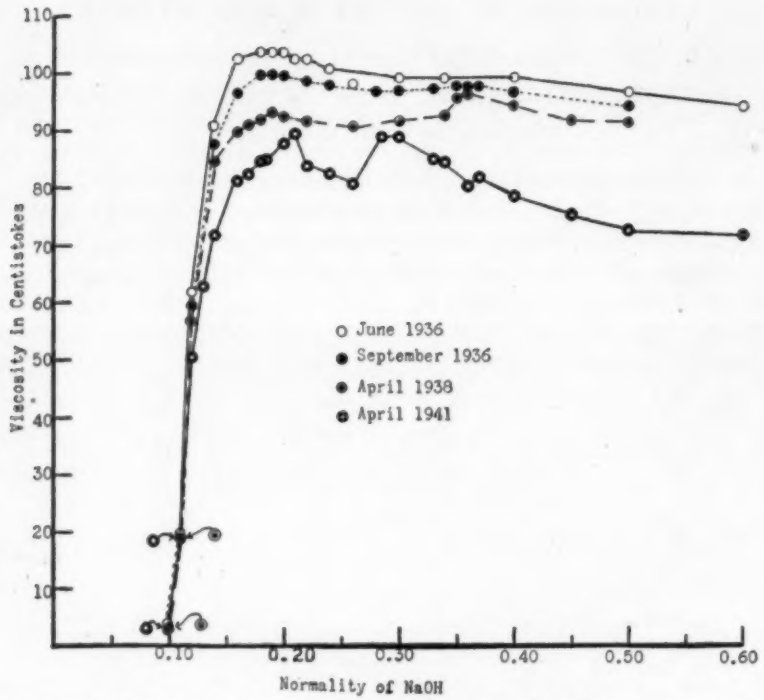


Fig. 1. Showing hysteresis in the viscometric behavior of a 2% "Thatcher" wheat starch sol cold gelatinized with various concentrations of NaOH at 30°C.

Four series of runs at different times are shown in Figure 1. It is evident from these curves that the starch sample has changed progressively with time, aging resulting in a marked lowering of the maximum viscosity which is attained.

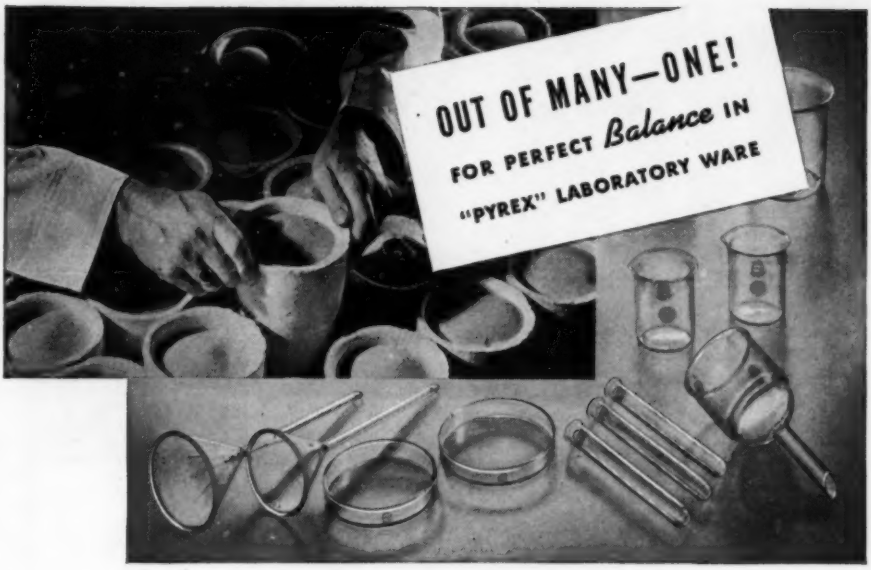
At present we are not theorizing on the form of the viscosity curves, but apparently aging results in a shifting of both the maxima, which originally occurred at approximately 0.18*N* and 0.35*N* NaOH. Aging apparently shifts the first maximum to a higher NaOH concentration and the second maximum to a lower NaOH concentration, and greatly accentuates the dip between the maxima.

Summary

Air-dry wheat starch undergoes hysteresis with time as evidenced by cold gelatinization viscosity behavior. An air-dry wheat starch cannot be used over a period of years as a viscometric standard.

Literature Cited

Ostwald, W., and Frankel, G.
1927 Kinetische Studien über Stärke-Verkleisterung. I. Über Kaltverkleisterung von Stärken. Kolloid Z. **43**: 296–312.
Ripperton, E. T.
1931 Measurement of consistency of starch solutions. Ind. Eng. Chem. (Anal. Ed.) **3**: 152–154.



During its many years of glass making, Corning Glass Works has developed more than 25,000 glass formulae. More than 250 different formulae are used in daily production.

Of all these formulae, none is more important to the laboratory than the formula used for PYREX brand Chemical Glass No. 774. For the development of this glass not only made America independent of foreign sources for heat-resistant laboratory ware, it did far more.

In 1915, for the first time, America had a *Balanced Glass* whose properties—mechanical and thermal strength and chemical resistance—were *scientifically balanced* for maximum laboratory usefulness, and economy through long life. Its balance gives to "PYREX" Laboratory Glassware the important qualities that make it the all-around glassware for all-around laboratory use.

When you buy laboratory ware, economize! Benefit from *Balance!* Standardize on PYREX brand Laboratory Glassware. Your regular laboratory supply dealer can furnish any of the thousands of items listed in Catalog LP21.

PYREX BRAND
LABORATORY WARE

PYREX — REG. U.S. PAT. OFF.

CORNING *means* Research in Glass

VYCOR — TRADE MARK REG. U.S. PAT. OFF.

"PYREX" and "VYCOR" are registered trade-marks and indicate manufacture by

CORNING GLASS WORKS
CORNING, N. Y.

INVESTIGATE *"Precision"* KJELDAHL

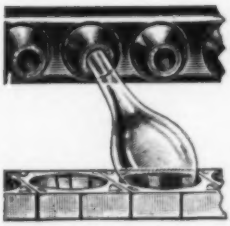
Before You Specify

IF you're going to put in Kjeldahl equipment, whether a portable unit or a roomful, don't complete the plan before you find out about the *most extensive* variety of Kjeldahl models now built. Standard or special assemblies to meet *any* lab requirement, *any* budget. Thousands of installations all over the world testify to superior performance.

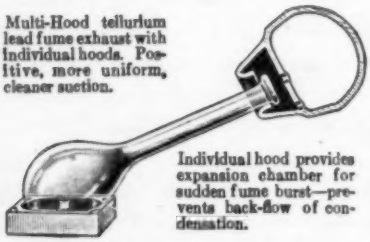
●

UPPER PHOTO—Shows only part of 72-unit Multi-Tube distillation at one of country's most prominent research centers.

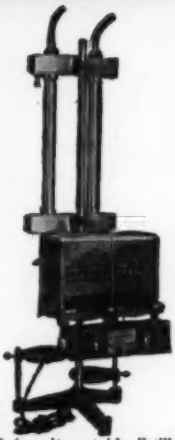
LOWER PHOTO—"Advance" model combination for digestions and distillations. Most modern Kjeldahl equipment built.



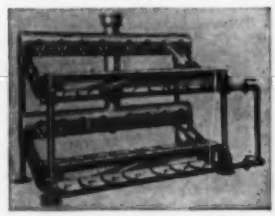
Multi-Hood tellurium lead fume exhaust with individual hoods. Positive, more uniform, cleaner suction.



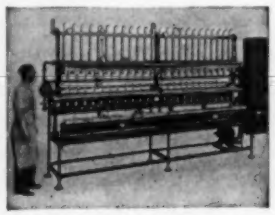
Individual hood provides expansion chamber for sudden fume burst—prevents back-flow of condensation.



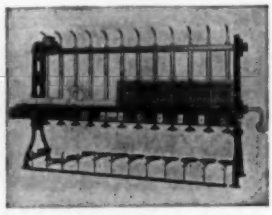
Twin-unit portable distillation, available in several models, gas or electric.



Double-deck Duriron digestion, gas heat. Open hole, nipple type, or for wet ashing.



24-unit combination, electric, rheostat panel-board. Any number of units possible.



Typical custom-built distillation outfit. Has 6 rheostat heaters.

SEND FOR SPECIFICATION BROCHURE

PRECISION SCIENTIFIC COMPANY

Designers and Builders of Modern Laboratory Equipment

1754 NORTH SPRINGFIELD AVE., CHICAGO, ILL.

CONTROL

UNBIASED laboratory tests over a period of several years have conclusively proved that NATIONAL GRAIN YEAST is far superior in the matter of dough control.

And it is because of this and other outstanding qualities that thousands of progressive bakers have come to regard NATIONAL GRAIN YEAST as a primary essential in the art of baking better bread.

NATIONAL GRAIN YEAST CORPORATION

Chanin Bldg., N. Y. C. • Chicago, Ill. • Crystal Lake, Ill. • Belleville, N. J

Frank J. Hale
President

★ Simpler
★ Faster
★ $220.00

Coleman
ELECTRONIC Photofluorometer

For every Fluorescence Technic, especially for the B_1 and B_2 determinations. ***Four times more sensitive and simpler! Use your own 18 mm. test tubes!*** A ONE-PURPOSE instrument for detection of small amounts. Base price $220.

The shutter device, suggested by Dr. J. S. Andrews, avoids destruction of test material. The built-in variable, electronic amplifier makes possible readings of extremely small amounts . . . especially required for Riboflavin.

Recent purchasers include Dr. Steenbock of U. of Wisconsin; Dr. Harrel of Pillsbury; Lt. Wodicka of Chicago Quartermaster Depot; Dr. Clifcorn of Continental Can Co.; Mr. Hennessy of Erwin Wasey Co.; Dr. Storms of Gerber Products; Prof. Rose of U. of Illinois; Dr. Sandstedt of U. of Nebraska.

★ Bulletin 205 is now off the press and also T108, technical comparison of five instruments, is available. We shall also be happy to send Bulletin 204 showing the COMBINATION UNIVERSAL Spectrophotometer. . . . For BOTH Transmission and Fluorometric methods.

Please mention your best priority rating.

★
Authorized Coleman Distributor
★

Laboratory Supplies and Chemicals

WILKENS-ANDERSON CO.

111 N. CANAL ST. CHICAGO

You CAN DEPEND UPON THESE TOP-NOTCH SHORTENINGS . . . . .

PRIMEX B&C

An all-hydrogenated vegetable oil shortening of exceptional stability. Excellent for all deep frying purposes. Preferred by biscuit and cracker bakers, manufacturers of prepared biscuit, pie crust, and doughnut flours, and makers of other food products where rancidity troubles are to be avoided.

SWEETEX

The "High-Ratio" shortening. Especially designed to permit bakers to produce "High-Ratio" cakes, icings, and sweet yeast goods with superior eating and keeping qualities.

PRIMEX

The all-hydrogenated shortening "that sets the standard." A quality shortening especially recommended for doughnut frying, for pies, cookies and bread, and for other shortening purposes.

PROCTER & GAMBLE

Branches and warehouses in principal cities

General offices - Cincinnati, Ohio

Tearing of Gluten Strands

and too much abuse of the gluten must be avoided in mechanical dough testing, it seems, if one wants to get a true picture of the gluten characteristics of a flour.

It is logical, too. The gluten must be given a chance to develop and swell, and not be broken down right from the start of the test by excessive abuse and tearing.

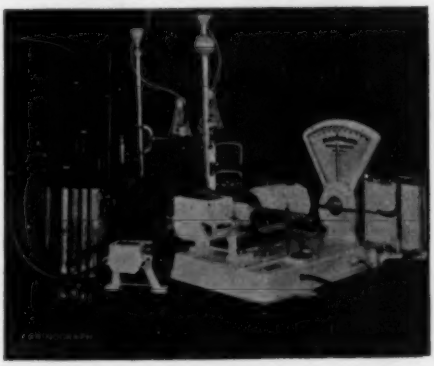
Of course, proponents of high-speed abusive dough testing have wanted to simulate high-speed mixing in bakeries. Good enough—but are mixing conditions similar between 50 grams of dough and a thousand pounds?

Decidedly not. The smaller the mass of dough, the greater the intensity of mixing and the abuse and tearing of the gluten.

Observe a baker's dough coming out of the mixer. It is well developed and smooth, but shows little actual tearing.

Especially important is this point in the testing of Soft Wheat flours. If a soft wheat flour gets too much abuse in a mixing test, the breakdown of its gluten overshadows its gluten development, and the curve leads to wrong conclusions.

These experiences have led to the development of the new double-speed FARINOGRAPH, with the high speed of 60 r.p.m. used for bread flours, and the slow speed of 30 r.p.m. for soft wheat flours.



YOU CAN HAVE ONE OF THESE LATEST MODEL, DOUBLE SPEED FARINOGRAPHS, WITH THE THREE-SENSITIVITY FEATURE AND TEMPERATURE-CONTROL THERMOSTAT, AGAINST A NOMINAL RENTAL CHARGE PER MONTH. WRITE US FOR DETAILS

BRABENDER CORPORATION, Rochelle Park, N. J.

Agene improves gluten characteristics and improved gluten means an improved loaf of increased volume, finer texture and better looking.

YEAR IN AND YEAR OUT "THE N-A MILLS ARE THE BUSY MILLS"

NOVADEL-AGENE

AGENTS: WALLACE & TIERNAN CO., INC., NEWARK, N. J.